MANUFACTURE OF PORTLAND CEMENT

BY

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PREFACE

THE Author's Lectures at the University of Cambridge upon the modern manufacture, testing and uses of cement, and those more recently delivered for the Royal Society and the Institution of Civil Engineers (and for which Mr. Davis was awarded the Mullins Silver Medal), have awakened a new interest in the science and technology of Portland Cement. I have thus decided to publish a third edition of the Author's book on the subject for the information of cement users.

The entire contents of the second edition have been re-written and brought up to date by the insertion of matter dealing with the latest available information concerning the manufacture and testing of Portland Cement, including a description of the newest labour saving devices and testing appliances and methods, and the latest theoretical conclusions as to its constitution.

The Author has taken pains to make the science of cement an easy subject for study by the consumer, and the valuable illustrations included in this new issue add additional interest and will further explain the text to those readers whose business it is to treat with Portland Cement.

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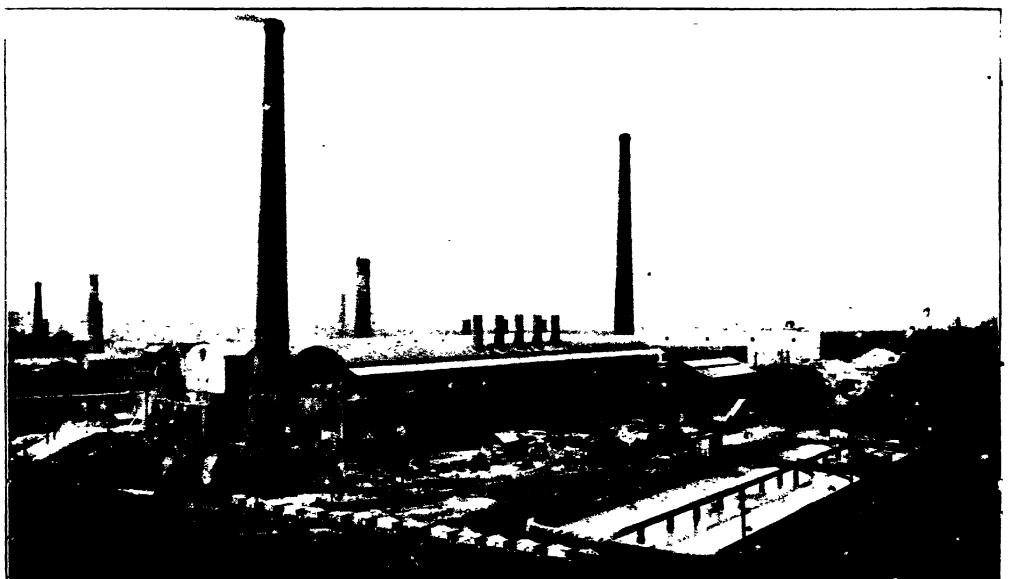
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THE LARGEST WET PROCESS CEMENT FACTORY IN ENGLAND

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THE MANUFACTURE OF PORTLAND CEMENT

Introduction.

CHAPTER I.

THE application of Portland cement in general construction covers so wide a sphere of usefulness that each year finds it successfully employed in some new work, and numerous instances of this point to the great strides which have been made during recent years in the science of the manufacture of this invaluable product. Foundations fortifications, bridges, breakwaters, docks, canals, dams, reservoirs, silos, cement-concrete streets, roads, and footpaths, with their remarkable neatness and evenness, together with partitions, roofing tiles, paving slabs, building blocks, boats, rafts, barges, caissons, and ships, and to-day, entire buildings, chimney shafts, and pressure pipe lines, all erected in reinforced concrete, represent a few of the important works carried out in Portland cement. These illustrations mark but the beginning of an era of cement in the field of engineering and architecture.

Originally invented in England about a century ago, Portland cement of British manufacture is still paramount.

For some years cement makers have been actively engaged in building new cement works and putting down up-to-date plant in obsolete factories, with the result that for quality of product combined with low cost of production, the British manufacturer has nothing to fear from oversea competitors, and British Portland cement to-day has no rival in quality the world over.

The debt which the community owes to Portland cement is hardly recognised as widely as it should be. Its inven-

tion alone rendered possible many of the extraordinary architectural, and more especially engineering, feats of the past century—its durability far exceeding iron, bricks and building stone.'

—The present excellence is the outcome of years of patient scientific investigation and study; and, although the names of the inventors and perfectors of Portland cement (who laboured principally during the first half of the nineteenth century) are forgotten, or, at least, but dimly remembered, the result of their work has originated and assisted design to as great or perhaps to a greater extent than has the introduction of mild steel for constructional purposes.

History of Cement—Smeaton, 1757. As to the history of cements, the first information to be found in regard to the discovery of Portland cement or its allied materials is in the records left by that eminent engineer, John Smeaton, who, during the construction of the first Eddystone Lighthouse, in 1757, discovered that by burning a mixture of lime and clay the resulting product possessed the property of hydraulicity. It is reasonable to suppose that the material which Smeaton then prepared was a hydraulic lime as we know it to-day.

Vicat, Fifty Years Later.—Little notice was, however, paid to Smeaton's discovery, and some fifty years later the French chemist Vicat followed Smeaton's observation to its natural and logical conclusion in making a cement by burning together finely pulverised chalk and clay, after having mixed them into paste. His process, however, again received little attention.

Aspdin in 1824. In the year 1824 the practical commencement of the manufacture of what we know to-day as "Portland" cement took place, when the English mason, Joseph Aspdin, of Leeds, found that by mixing in certain well-defined proportions finely pulverised chalk with clay and burning the same at a high temperature and grinding the product, he was able to produce a hydraulic binding material far exceeding in quality any product known up to that time. This material possessed the delicate grey colour of Portland stone, and

thus received the name of "Portland" cement, which formed the subject of Aspdin's oft-quoted patent of October 21st, 1824.

First used in Thames Tunnel, 1828.—It is safe to assume that one of the Thames tunnels, constructed in 1828, was the first engineering work of importance in which Portland cement was used. The price at that time was 21s. per cask at the works, and a superior quality product could be purchased prior to the war at some 8s. per cask, so much have the conditions of the industry altered in the past three-quarters of a century.

What Portland Cement is.—The term "Portland" is limited to a hydraulic cement containing proportions of lime, silica and alumina within certain limits, and at once distinguishes the valuable constructive material bearing that title from other cementitious substances.

As the classifications of hydraulic products vary, so do the definitions of Portland cement, and the opinions as to what shall be included in the term.

To be perfectly clear, then, as to what Portland cement actually is, reference should be made to the official descriptions of the product in the largest cement-consuming countries, viz.: -

Great Britain.—The British Standard Specification defines Portland cement in the following terms:—"The cement shall be manufactured by intimately mixing together calcareous and argillaceous materials, burning them at a clinkering temperature, and grinding the resulting clinker, so as to produce a cement capable of complying with the specification."

France.—In the French Government Specification Portland cement is defined as:—"The product of the grinding of clinkered rock, obtained by burning to the point of softening an intimate mixture of carbonate of lime and argillaceous matter, rigorously combined and chemically and physically homogeneous in all its parts."

United States of America.—The American Standard of Portland cement defines the product as:—"The finely pulverised product resulting from the calcination of an intimate mixture of properly proportioned argillaceous and

calcareous materials, and to which no addition greater than 3 per cent. has been made subsequent to calcination."

Summary of Descriptions.—So it will be seen that from a study of these various descriptions we really arrive at the point that :—

Portland cement is a chemical product obtained by the preliminary mechanical mixture of carbonate of lime with silica and alumina, which after passing through the succeeding stages of manufacture, results in a combination of silicates and aluminates of lime.

The centres of the manufacture of Portland cement are well distributed over the country—namely, in the North and South, Midlands, and East of England, and Wales in the West. In Ireland, the latest cement factory has been erected near Larne. In Scotland, cement works are situated near Glasgow and Edinburgh. There are to-day about 60 factories in England, 4 in Wales, 4 in Scotland, and 2 in Ireland.

The normal size of a factory is one having an output of some 500 to 1,000 tons of Portland cement per week, and in the United Kingdom alone there are cement factories with a total output of 3,500,000 tons of cement per annum.

The Capital Cost of these factories, upon a broad estimate of £4 per ton of annual output, would approach some £14,000,000.

The number of Employees in the industry, based upon a figure of six tons of cement output per man per week, approximates 12,000 men.

The extent of the Industry over the World may be gauged from the following details of total manufacture in the largest cement producing countries, viz. :—

	Tons per Annum.
United Kingdom	3,500,000
U.S. of America	16,000,000
Germany	6,000,000
France	1,250,000
Canada	1,000,000
Italy	1,000,000
Belgium	1,000,000



View of a Modern Dry Process Cement Works with Shaft Kilns.

Portland cement can be produced from any raw materials containing constituents capable of yielding by calcination the silicates and aluminates of lime which form its chief components; and the necessary constituents of these raw materials are lime, silica and alumina. Small additions of iron oxide are desirable for fluxing those materials.

The raw materials used for the manufacture, therefore, are carbonate of lime, usually in the form of chalk or limestone, and silica and alumina, usually in the combined form of shale or clay.

The two materials for the necessary combination indispensable for the manufacture of cement are not always found upon the same site. For the Thames and Medway works, as an instance, a chalk deposit, usually flinty, is found on the site where the works are situated, and this, together with clay or mud taken from the banks of the rivers Thames and Medway, is used in the manufacture. On the other hand, the Cambridgeshire and Bedfordshire works are able to obtain from one deposit all the necessary raw materials for the manufacture of Portland cement. Works in the Rugby and South Wales districts use limestone and shale from the geological deposit known as Lias. On the Humber, chalk is used, and in Ireland an indurated chalk with clay dredged from Lough Larne is used. In Scotland, blast-furnace slag is used as a raw material, and at other works hard limestone is employed.

The suitability of locality depends primarily upon the manner of the occurrence of these requisites, the location of the deposit with respect to the cement-consuming market and the fuel supplies, for since with every ton of cement manufactured there will be used half a ton of coal, the location of the factory in regard to cheap fuel supplies is quite an important factor.

If it were at all possible to find a geological formation of uniform composition containing exactly the right amount of naturally mixed chalk and clay, Portland cement could be made from it by the cheap and simple method of merely burning lumps of the material as quarried and grinding the resultant clinker, but since such a material has not been discovered in quantity in Great Britain, this method of manufacture is hardly practicable, for a variation of even 1 per cent. of the proportion of car-

bonate of lime in the raw materials, to say nothing of the irregular composition of the other ingredients is sufficient to destroy the reliable quality of the resultant cement.

In manufacturing Portland cement, carbonate of lime and clay, or other argillaceous material, are first efficiently and accurately amalgamated in certain fixed proportions, either with the addition of water to the raw materials or by the fine grinding and mixing of the same in their dry state.

The composition of a prepared mixture of these raw materials should be, broadly, three-parts of chalk, or calcareous constituent, to one of clay, or argillaceous constituent.

The various raw materials commonly employed in the manufacture of Portland cement at the present day may be more fully described as follows:—

Thames and Medway Materials.—In the Thames and Medway districts the chalk formation on the banks of the rivers is quarried, and this material is mixed with the deposits of clay found in the estuaries and creeks. The chalk deposits, often containing flints, which must be extracted, have a depth of 100 to 300 feet, and the excavation is carried on by mechanical means. The greater part of the clay used by the Thames and Medway manufacturers is the alluvial mud referred to, forming the beds of the rivers. The two raw materials are mixed together in correct proportions by what is called the **wet process** of manufacture, which will be explained in another chapter.

Cambridgeshire Chalk Marls.—The chalk marls around Cambridge have long been proved to be suitable for the manufacture of cement. The "marl" is a naturally mixed deposit of calcareous and argillaceous material and is to be found at the base of the local chalk. It differs from the main mass of the chalk above in the quantity of the clay it contains, as might have been expected, seeing that the chalk deposit succeeded geologically a deposit of fine gault clay, and it is probable that during the changing geological conditions the sea would not be at once cleared of mud.

Lias Formation.—In the cement-making districts of Warwickshire and South Wales the Lias formations of limestone and shale are employed, and these materials are most successfully treated for the manufacture of Portland cement. The thin layers of limestone, with their variation in composition, must be very accurately and efficiently dealt with. In working the Lias formation it is found that the preponderance of shale in proportion to



Quarry in the Lower Lias Formation.

limestone (which is generally found to contain 78 to 85 per cent. of carbonate of lime) renders the cost of working heavy, because of the removal of surplus clayey material not required in the manufacture.

The Lias formation consists of layers of impure limestone of varying thickness, ranging from 2 in. to 2 ft., and parted by bands of shale and clay, the total thickness

of shale beds, considerably exceeding, as a rule, that of the limestone, which necessitates removal and tipping the waste.

Northern Works and Materials.—On the Tyne, Tees and Humber and in the North of England generally, Portland cement is sometimes manufactured from chalk on the spot or imported from the Thames, Medway or Sussex districts, and this is mixed with a local clay found on the site of the works. From this mixture a good quality cement is manufactured.

Scotland.—Near Edinburgh pure limestone is mixed with lower grades of stone. Other Scottish works use slag as a raw material.

Ireland.—Near Larne, a deposit of indurated chalk (or limestone) is employed for the manufacture of cement, and this is intimately mixed with clay dredged from the Larne Lough.

The above descriptions deal with the most important deposits of cement raw materials over the country.

In the United States most of the cement produced is from the Lehigh Valley argillaceous limestone, which contains rather more clay than is required for a correct mixture. To this a small amount of pure limestone, usually 10 to 20 per cent., is added to bring the mixture up to the necessary percentage of calcium carbonate required in the manufacture.

Summary of Raw Materials.—It will thus be seen that, given chalk and a suitable clay in approximate proportions of 3 to 1, or lime with silica and alumina in any other form, but correctly proportioned, it is of the first importance that these materials shall be treated with a full knowledge of the all-important chemical and mechanical operations of combining them, which alone can secure the manufacture of a reliable product.

Raw Material Mixing.—The mixing processes by which these raw materials are brought to the condition for chemical combination will be duly explained in detail, but it should be emphasised that the preliminary mechanical blending of the raw materials for the manufacture of

Portland cement is a stage requiring the utmost technical skill. The thoroughness of the process primarily determines the quality of the resulting cement, and if the manufacturer is to turn out a product of reliable quality, the scientific supervision of this branch of the manufacture is of paramount importance.

The first conditions of any method of preparing raw materials for Portland cement manufacture are—that they shall be (1) correctly proportioned, (2) very finely comminuted, and (3) thoroughly mixed.

The proportions of chalk and clay must be kept to a standard as close as possible to the ascertained analysis, and this at the present time, in the most successful cement works, is carried out under the supervision of the works' chemists.

It is, firstly, essential that extreme care should be exercised in obtaining the correct proportions of the chalk and clay, or whatever other raw materials may be used in the process, so that the resultant mixture may be relied upon to contain the exact chemical constituents necessary for the manufacture of a thoroughly sound cement; for, if the raw materials are not mixed in this proper proportion, nothing can be done later to correct it, and the result will be an inferior product.

The proportioning of the ingredients is governed by continuous laboratory checking of the raw materials as they are quarried, and this testing by the works' chemists goes on night and day, as long as the manufacture is proceeding.

How the next preliminary condition of the manufacture can best be fulfilled—that of the reduction of the raw materials—depends chiefly upon their character, so that the details of the methods adopted by manufacturers for grinding generally vary with the nature of the raw materials used.

Two Processes of Mixing.—There are two principal methods of reducing and mixing the raw materials: firstly, by the "Wet Process," applicable mainly, although not solely, to soft materials, the correct quantities of the raw ingredients being ground and mixed by the aid of a considerable amount of water; secondly, the "Dry Process," in which the perfectly dry materials are ground

together to an impalpable powder or "flour," and subsequently mixed to correct chemical proportions.

Wet Process.—The wet process of mixing the raw materials is particularly adapted to such materials as are easily disintegrated by the addition of water. This method of mixing, as originated and developed in the United Kingdom, is employed to a considerable extent by the manufacturers of cement on the Thames and Medway, which is still the chief seat of the industry. By this process the soft chalk, as quarried, and clay are weighed into what is called a "wash-mill," where a large amount of water is added. The wash-mill is usually a circular brick-lined pit, often of some 15 feet or more in diameter, and 5 feet deep. In the centre of this pit is a concrete or brick pier, carrying a vertical shaft, driven through a crown-wheel and pinion. The vertical shaft carries a circular framework, from which are suspended harrows fitted with steel tines. The passage of these tines through the chalk and clay suspended in water tends to break up the lumps, and convert the mixture into a liquid of thick, creamy consistency, which is kept in the mill until it is fine enough to pass the screens covering the outlet ports. The product of the wash-mill is termed slurry or slip.

In some instances the further reduction of the raw materials is effected in wash-mills alone with fine screens, while with some harder materials it is necessary for the slurry to be treated finally in the tube-mill, or put through a separating mill, while with hard materials crushers are required, and ball and tube-mills are used with water for fine grinding.

Slurry from a wash-mill plant contains about 40 per cent. of water, and when finally ground leaves a residue of less than 5 per cent. of its solid constituents on a sieve of 32,400 meshes per square inch.

Wet Tube-Mills.—The tube-mill, as used for wet grinding, consists of a drum (about 20 ft. long and 6 ft. diameter) made of steel, and protected against wear and tear by renewable cast-iron plates. The drum is nearly half-full of flint stones, and its interior is easy of access through manholes. The raw mixture previously reduced by the wash-mill flows through a hollow journal into the

revolving grinding drum. After being thoroughly ground by the flint stones, the material is discharged through the hollow journal at the opposite end.

Thick Slurry Process.—In the latest practice the wet process is somewhat modified to allow of harder materials than chalk and clay being treated thereby. This modification is known as the "Thick Slurry" process, and the raw materials are first of all crushed to a size not exceeding a 2-inch cube, then passed into a mill of the ball-mill type—*i.e.*, a revolving drum containing steel balls up to 5 inches diameter, together with a stream of water. The partially ground slurry issuing from the ball-mill is then treated by a tube-mill, where the final reduction occurs.

"Thick slurry" contains about 32 per cent. of water, and is usually even more finely ground than wash-mill slurry.

Dry Process.—In the dry process method of dealing with the raw materials for cement manufacture, the calcareous and argillaceous materials, of whatever substances they may be composed, have first to be dried after passing the preliminary crushing machinery. The dryers often take the shape of expensively constructed brickwork kilns, but, with the more general adoption of the dry process of manufacture, little time was lost in inventing a less costly and more efficient plant, which could also be worked at a much reduced labour cost. The dryers now usually employed, therefore, in the most modern plants consist of revolving iron cylinders or drums, some 30 to 50 feet in length and about 4 feet in diameter. These rotary drying drums are supported on steel tyres resting on heavy friction rolls, and the drums are rotated when in use at a speed approximating two revolutions per minute, and are usually set with an inclination of about one-half inch per foot.

The raw materials after passing the crushing rolls are introduced into the upper end of the dryers, and are immediately caught by cascading channels fitted inside the drums, which lift and drop the crushed raw materials as the drum revolves, and present them to the hot gases passing through the drums in the opposite direction to the way

the materials are travelling to the lower or outlet end. The waste heat from the kilns is thus employed for drying purposes; or, again, furnaces are sometimes arranged to rotary dryers for the external heating of the drum.

Dry Grinding and Mixing Raw Materials.—The raw material, after being crushed and dried, must then be reduced to an extremely fine powder or "flour" by such plant as is adopted for clinker grinding (and which will be explained later) and then carefully and thoroughly mixed to the proper chemical proportions before being conveyed to the kilns for burning.

Materials Necessitating Dry Process.—This dry process of manufacture has been employed where the raw materials cannot be satisfactorily reduced by wash-mill plant. It has been adopted in the Rugby and Wales districts and in the Cambridge district also, but is now, to a large extent, being superseded by the thick slurry process.

Processes in America.—In America and Canada the dry process is generally adopted, and in other countries extensively producing cement the dry process of manufacture is considered a less costly method than the wet process, but this again depends upon many local features.

Preparing Raw Materials.—It will be seen, therefore, that in preparing the raw materials for the manufacture of cement, the chalk and clay or other materials of similar composition must first be reduced to the utmost fineness, either by the addition of water or by dry grinding, either of which processes breaks down the cohesion between the particles, and leaves the material in a very finely divided state, the physical properties of the respective materials to be dealt with generally determining which method of reduction is to be adopted.

Final Composition of Raw Materials.—Before the prepared raw materials pass to the next stage in the manufacture—that of burning—the composition of the raw material mixture is ascertained by analyses and tests, and the carbonate of lime (familiarly known by its chemical formula, $CaCO_3$) should be kept within, at most, one-half per cent. of the quantity found to produce the best cement. This percentage varies slightly in different works, accord-

ing to the many geological formations contributing to the manufacture, but, broadly, a combination of 75 per cent. of chalk and 25 per cent. of clay will produce a cement of good quality. If any carelessness is permitted in the mixing process the results are likely to be disappointing, for when the proportion of clay runs too high (or, say, the percentage of lime carbonate falls below 75) a compound is obtained which, in the burning process, will fuse at a temperature lower than that required for the production of sound clinker, thus rendering it comparatively useless. On the other hand, an excess of chalk (above, say, 78 per cent.) will allow a mixture to sustain the highest temperature in the kiln without risk of fusion, but the resultant clinker would be of doubtful quality because of its high-liming or expansive tendencies when ground for cement.

Again, a variation in the amount of carbonate of lime, even to one-half per cent., is found to alter appreciably the tensile strength of a cement—namely, high lime increasing the strength, and low lime producing cement of little strength.

Analysis of Mixed Raw Materials.—The chemically mixed raw material prepared in the ordinary course of manufacture may, therefore, analyse in this way:—

Silica	...	16.5 per cent.	
Alumina and oxide of iron	...	6.5 ,,	Clay.
Undetermined ...	...	0.5 ,,	
Carbonate of lime ...	...	75.5 ,,	
Carbonate of magnesia		1.0 ,,	Chalk.

From such a material, if properly treated in the further stages of manufacture, a good commercial cement, testing (neat) at least 650 lbs. per square inch in seven days, should be produced. The raw materials after combination are then carefully mixed and proved before proceeding with the manufacture.

Burning or Calcining.—The next stage in the manufacture of Portland cement, following the scientific and mechanical preparation of the raw material, is that of burning at a high temperature, or calcining, the raw product to the point of slight vitrifaction, resulting in what is commonly called a cement "clinker."

As will be seen from the foregoing description, Portland

cement consists of a chemical combination of lime, silica and alumina, and these materials are combined together under the action of great heat, thus becoming a mixture of silicates and aluminates of lime. The resultant material is ground to an impalpable powder, after which the product is ready for the market.

Silicates and Aluminates of Lime.—In the raw materials it will be observed that there is no combination between the carbonate of lime on the one hand and the silicate of alumina on the other hand. In the conversion of the prepared raw materials into cement clinker by burning, the silica and alumina of the clay immediately enter into combination with the lime, thus forming calcium silicates and aluminates. These compounds are the important constituents of a Portland cement, and give to it, when combined in their proper proportions, its hydraulic properties.

Impurities in Raw Materials. None of the cements of commerce, however, are made up wholly of these three ingredients, for the raw materials from which cement is made are never quite pure. It has been found, for instance, that iron oxide behaves, in burning a mixture, the same as alumina, and that a good cement could be made in which all the alumina was replaced by iron oxide, but the essential elements of a good cement are that it shall contain at least the necessary amount of silica and lime.

Heat Regulation.—The calcining process is a purely chemical one, and it is a stage of the manufacture involving great responsibility; for, just as in the primary blending of the chalk and clay, a faulty mixing can make or mar the quality of the cement, so can the deficient burning of the material destroy all that is valuable in the finished product. If the heat be not sufficient, the necessary chemical changes do not take place, and an equally unsatisfactory result is obtained.

Degree of Burning.—The proper degree of burning is indicated by the formation of a dense greenish-black clinker. Light-burned clinker is yellow and soft, while over-burned clinker is fused and slag-like. Thermometric measurements made during the process of calcination

show that for normal Portland cement burning a temperature is required in the kiln amounting to about 1,400° Centigrade, or 2,500° Fahrenheit. This temperature is variable, according to the percentage of carbonate of lime contained in the raw materials—that is to say, the higher the proportion of lime the higher the temperature which is necessary to produce complete diffusion in combination with silica. Iron oxide also has the effect of reducing the clinkering temperature.

Types of Kilns.—The kilns for burning the raw materials are constructed for either an "intermittent" or a "continuous" process. The intermittent kilns, now rarely adopted, are those employed in the wet process, and are of such a design that the burning necessitates distinct loading and drawing operations, giving intermittent working, and demanding the complete cessation of the burning process during the drawing operation. Since this method of manufacture is one that soon will be discarded, it is not necessary to devote much time to an explanation of working this type of kiln.

Continuous Kiln—In the continuous kilns, the burning of the raw material proceeds without a break, and the drawing of clinker takes place at the same time without interruption, thus making a continuous process.

Fuel Economy with Continuous Kiln.—Continuous kilns are generally costly to construct, and require skilled labour for their operation. These kilns are economical in fuel, and where they are used the calcining process is much more regular, and the cement is of better quality.

The Rotary Kiln.—In the category of continuous kilns is included the "rotary" kiln, which is the most modern appliance for calcination and the most widely adopted. The manufacture of cement by the rotary kiln may be said to have revolutionised the industry during the past twenty years, and this method of burning the raw materials is, perhaps, the most scientific and practical invention that has been introduced into the manufacture since Portland cement was first known. Although the use of this kiln is a technical and expensive process,

involving heavy capital outlay and a high cost of production and continual upkeep, there is nothing yet in sight to outclass this invention in the manufacture of Portland cement as carried on to-day.

Rotary Kilns in U.S.A. and Europe.—More than 90 per cent. of the cement manufactured in the United States is produced by the rotary kiln; and in Europe in the majority of cement works this kiln is solely adopted.

Quality of Rotary Kiln Cement.—Although the rotary kiln was the subject of much doubt and prejudice when first introduced, it is safe to say that no new cement factory would be erected in the present day with any other type, while it is probable that 90 per cent. of the cement used in the United Kingdom is the product of this kiln.

The kiln consists of a slightly inclined steel or wrought-iron cylinder, usually from 100 to 200 feet in length, and 6 to 9 feet in diameter, and is inclined to the horizontal at about 1 in 30. The size of the kiln within these limits determines the quantity of the output—a rotary kiln 200 feet long and 9 feet diameter producing 1,200 tons of clinker a week, with night and day running. The rotary kiln is lined with radial firebricks, 6 to 9 inches in thickness, and the long cylinder is mounted on tyres running on rollers, and is slowly rotated by gearing.

Rotary Process of Burning.—The cement-making materials, after due preparation, are continuously fed into the kiln through a pipe at the upper end in the form of either a liquid (slurry) or dry powder, according to the process adopted in preparing and mixing the raw materials. Finely ground coal is almost always used as fuel, and this is introduced into the lower or outlet end of the kiln by a jet of air issuing from a blast fan.

Fuel Ignition.—When the kiln is started the fine coal is ignited, and after a time a white heat is obtained in the lower end of the cylinder. The raw material is then fed into the kiln, and, as it gradually descends into the zone of heat generated by the perfect combustion of the finely ground coal fed into the cylinder from the

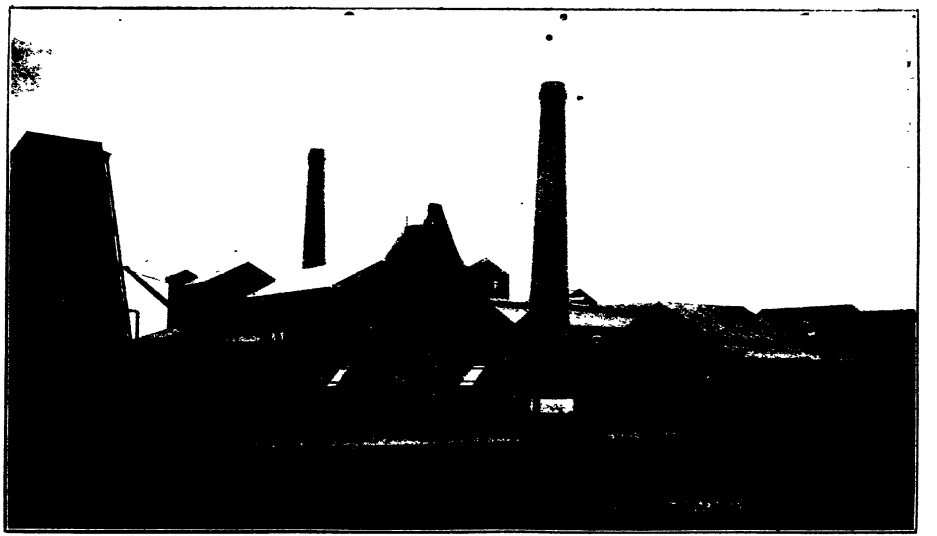
opposite end, it parts with its carbonic acid, forms little balls which reach a nearly white heat in the lower third of the kiln, and finally issues at the lower end as well-burned clinker in grains up to about the size of a large pea. The greatest heat is naturally near the fuel jet, or outlet end of the kiln.

The operation of calcining is a continuous one, and with proper care, under- or over-burning may be avoided. The hot clinker is cooled by passing through rotary cooling drums, similar in form to the rotary kiln, but much smaller in size.

Cooling Drums.—The cooling drums now generally in use in this country are placed at the lower end of the kiln, and receive the hot clinker as it drops finally from the kiln. Coolers generally consist of steel tubes some 40 feet long and 4 feet in diameter, containing cascading channels for lifting and dropping the hot clinker as the coolers rotate, thus presenting it to the cold air passing through the cooler on its way to the kiln. They rotate at a somewhat higher speed than the furnace tube, and by the air for the latter being drawn through the red-hot clinker a fair proportion of the otherwise lost heat is retained and utilised. When the clinker issues from the coolers it is quite cold enough to handle, and to pass to the further process of grinding into powder.

But before this stage it is necessary to point out that all rotary kiln plants with ground coal firing must include an apparatus for the drying of the coal to be used in the burning, and also a grinding plant to enable the coal to be finely pulverised before entering the kilns.

Description of Coal Dryer.—The most common design of coal dryer consists of an inclined steel cylinder encased in brickwork. The hot gases from the fires of the coal dryer or from the clinker cooler, coming first in contact with the cylinder at the point where the wet coal is introduced, pass along and around the tube on the way to the chimney-stack, which is located over the discharge end, whilst a separate chimney at the feed-end of the rotary coal dryer permits the steam from the moist coal to escape, and gives an outlet for any accumulating gases or coal dust.



MODERN PORTLAND CEMENT FACTORY.—ROTARY PROCESS.

Operation of Working.—The wet coal is introduced at one end of the heated drying drum, and by means of cascading channels inside, and the slow rotation of the inclined cylinder, the coal passes through the drum, and by the time it arrives at the outlet end the dried coal is quite suitable for the purpose of fine grinding for use in the rotary kiln.

Coal Grinding and Delivery.—From the dryer the coal falls into a conveyor, and is carried automatically to the coal-grinding mill, which contains machinery for reducing the dried small coal to an impalpable flour. For the process of coal-grinding the usual ball and tube mills, or Griffin mills, are required, and this plant is, therefore, similar to the clinker-grinding plant, particulars of which will be given in a later chapter. The finely ground coal is then taken to the kilnhouse, where it enters the coal-hoppers placed in front of each rotary kiln, and from these hoppers an automatic feed conveys the powdered coal to a point where it is met by the heated air blast from the fan to the kiln. When reaching the heat of the kiln the powdered coal immediately bursts into flame, and the intense heat in the kiln is thus kept continuously regular by the constant coal feed.

Percentage of Fuel.—The quantity of fuel used in burning cement by the rotary kiln is from 25 to 35 per cent. of the clinker output, and these percentages vary considerably according to the process of manufacture, the quality of coal used, the fineness of grinding, and the facility with which the raw materials are burned.

Quality of Coal.—A satisfactory coal for the rotary kiln is generally obtained from the various bituminous coals found in the United Kingdom, but it is desirable that the percentage of ash in the coal should not exceed 10, and that the coal should have a calorific value of some 12,600 British thermal units per lb. of coal.

Clinker Grinding.—To proceed with the manufacture of Portland cement, and in arriving at the succeeding and final stage in the process, we have the grinding into an extremely fine powder of the clinker which comes from the kilns. This process has exercised quite a large proportion of ingenuity during the past few years.

So much attention has of late been devoted to the manufacture that, as regards the mixing of the raw materials in their due proportion and the proper calcination of the mixture so obtained, one brand of cement may be said to be very much like another, provided the works are suitably designed and the various details of the manufacture are carried out with care.

Quality and Fineness of Cement.—The question then arises—What constitutes a high-class cement? To which question the answer is—That the true test of quality is in the fineness to which the cement is ground—always provided, of course, that the materials are carefully selected and the various stages of manufacture are properly carried out.

It is not intended, however, that the scientific qualities of cement produced by grinding to various degrees of fineness should be here dealt with. I propose now merely to describe the machinery generally employed in the process of clinker reduction. It must be explained, however, that in clinker grinding not only is it necessary that a certain degree of fineness be attained to make a satisfactory quality cement, but that also as large a proportion of "flour" as possible shall be contained in the finished product. By "flour" is meant the extremely fine particles of cement in which the cementitious property of the material is believed to reside principally, as apart from the "residue," which consists of practically inert material. These are terms which, as is known, are used in the testing of cement for fineness.

Clinker Crushing.—With lumpy clinker the first stage in its reduction is generally carried out by an ordinary stone-breaker, or crusher, or by rolls, which reduce any large lumps to sizes from about ¾-inch cube down to coarse dust, and after this operation the clinker is conveyed to the fine grinding machinery.

Ball Mill.—In modern works the preliminary grinding of cement clinker is carried out by the ball-mill, and from this mill the coarsely ground material is conducted to a tube-mill which finishes the fine grinding previous to storing the cement. The ball-mill consists of a cylindrical grinding drum, mounted on a steel shaft running through

it, and provided with a tightly closed sheet-iron casing. The grinding drum is composed of overlapping steel grinding plates, in one-half of which are holes for the ground material to find its way through to the fine sieves externally surrounding the drum, and through which the somewhat coarsely ground cement passes, and is conveyed to the tube-mills for finer grinding. The crushing action of the ball-mill is caused by the revolving of the drum, which contains a number of steel balls of various sizes between which the clinker is crushed and pulverised.

Tube Mill.—For the finishing process—the fine grinding proper—the tube-mill is employed, which grinds by means of the round flint stones or steel pellets contained within it.

The tube-mill consists of a wrought-iron revolving cylinder, with hollowed pivots at both the feed and delivery bearings, and is about one-half full of rounded flint stones or other grinding medium. The coarsely ground cement is fed from the ball-mill into one end of the tube-mill, and the rotatory action of this mill, similarly to the ball-mill, finely pulverises the cement as it passes through the falling flint stones to the delivery end.

Degree of Fineness.—The finished cement is ground sufficiently fine to pass through a 180 x 180 mesh sieve with about 10 per cent. residue, and finer grinding can be readily accomplished by the manufacturer if necessary; but this means a reduced output from the plant.

Stores.—From the grinding mills the cement is conveyed into the stores, and after it has cooled down the material is ready for loading out at the factory.

As to the storage of Portland cement, it is generally considered that the longer the cement is kept in stock the more reliable it is found to be in use; but the modern product of to-day's manufacture requires neither storage nor aeration to provide and ensure the success of its quality.

Aeration of Cements.—With the product manufactured years ago it may have been necessary to aerate cement for some considerable time before introducing the same into the work, but with the cements manufactured

o-day this is quite unnecessary. The aeration of cement s a safeguarding process adopted by engineers with important work, and was, perhaps, then advisable to liminate any "free lime" that might have been contained in the material, as an underburnt or carelessly manufactured cement would contain a considerable quantity. Cement such as this would, when mixed with vater, set quickly, and sooner or later the particles of 'free lime" absorb moisture, and change from what s termed "quick lime" (CaO) to a hydrate of lime $Ca(HO)_2$, and the expansion which accompanies this hange produces ominous cracks in the hardened cement.

The old idea was, therefore, that if the cement be exposed to the atmosphere for some time before using, any ree lime which it may contain will absorb moisture from he atmosphere and become slaked, and will not then be iable to cause injury to the concrete after setting. But cement made from accurately mixed raw materials, well-burnt and finely ground, as all good cement should be, will not require any aerating process, and as soon as the cement is manufactured it should withstand the recognised tests for quality.

Cooling.— It is desirable, however, that the material hould be cooled down before loading into bags, and, therefore, the manufacturer generally arranges that he has sufficient quantity of material in stock to provide that engineers are supplied with a perfectly cool cement for their work.

The Composition of Portland Cement.

CHAPTER II.

HAVING given in brief outline a definition of Portland cement as is generally accepted to-day, and also a description of the manufacture as usually adopted in the United Kingdom, it may now be necessary to give a resumé of our present knowledge and the existing theories as to the chemical constitution of Portland cement.

In this respect there are two outstanding features of prime importance to consider in relation to the properties of the raw materials which are used for the manufacture of Portland cement. These are known as the "Lime Ratio" and the "Silica Ratio."

The tests for the chemical composition of cement are defined in the British Standard Specification as follows, viz. :—"The proportion of lime, after deduction of the proportion necessary to combine with the sulphuric anhydride present, to silica and alumina when calculated (in chemical equivalents) by the formula,

$$\frac{CaO}{SiO_2 + Al_2O_3}$$

shall not be greater than 2.85 nor less than 2.0. The percentage of insoluble residue shall not exceed 1.5 per cent.; that of magnesia shall not exceed 3 per cent.; and the total sulphur content calculated as sulphuric anhydride (SO_3) shall not exceed 2.75 per cent. The total loss on ignition shall not exceed 3 per cent."

For instance, in the case of a cement containing 63.28 per cent. of lime, 21.6 per cent. of silica and 8.16 per cent. of alumina, and 2.00 per cent. of sulphuric anhydride, the proportion of lime, after deduction of the proportion necessary to combine with the sulphuric anhydride

present, to silica and alumina will be found to be as follows :—

Molecular weight of lime 56
" " silica = 60
" " alumina 102
" " sulphuric anhydride = 80

Lime combining with 2.00 per cent. of sulphuric anhydride $= \frac{2.00 \times 56}{80} = 1.40$ per cent.

$63.28 - 1.40 = 61.88$ per cent. Lime.

Lime	(CaO)	$= \frac{61.88}{56}$	=	1.10
Silica	(SiO_2)	$= \frac{21.6}{60}$	=	0.36
Alumina	(Al_2O_3)	$\frac{8.16}{102}$		0.08

Then $\frac{CaO}{SiO_2 + Al_2O_3} = \frac{1.10}{0.36 + 0.08} = 2.50$

The **Lime Ratio** determines the proportion (in chemical equivalents) between the lime on the one side and the silica and alumina on the other, and the idea underlying the limits imposed by the British Standard Specification (viz., 2.0 to 2.85) is that a true Portland cement must contain at least 2 molecules and not more than 2.85 molecules of lime to one molecule of silica, and one molecule of alumina. These limits have an empirical basis, and the lower limit serves to exclude slag cements, or those cements produced from slag without passing through the process of manufacture defined in the British Standard Specification, while the upper limit is a safeguard against an excess of lime, which tends to unsoundness.

In ordinary practice the limits for the production of good cement are 2.4 to 2.8, and commercial cements usually have a lime ratio not far from 2.6. If the lime ratio is below 2.4 a low tensile strain may be expected, and if the lime ratio is above 2.8 high expansion tests often occur.

The **Silica Ratio** expresses the actual proportion (not in chemical equivalents) between the percentage of silica

and the sum of the percentages of alumina and iron oxide, viz. :—

$$\text{Silica Ratio} = \frac{\text{Percentage of Silica}}{\text{Percentage of Alumina} + \text{Percentage of Iron Oxide}}$$

The silica ratio has a connection not only with the quality of the materials but also with its behaviour while in the kiln. Dealing first with the quality : a high silica ratio (*i.e.*, above 2.7) tends to produce a slow hardening cement, especially when the raw materials are calcined in rotary kilns, and unless the silica is in a fine state of division there is also a tendency to unsoundness and low tensile strength when mixed with sand, both defects arising from incomplete combination between lime and silica.

On the other hand, a low silica ratio tends to produce a quick hardening cement which develops its strength at an early date, so that the increase in tensile strength from 7 days to 28 days demanded by the British Standard Specification may frequently not be obtained.

With regard to the stage of burning to the point of incipient fusion the prepared raw materials in the kiln, materials with a high silica ratio can only be "clinkered" as it is technically termed at a very high temperature, and this involves undue wear and tear on the brickwork lining of rotary kilns.

On the other hand, materials with a low silica ratio - say, below 2.3—more readily clinker, and thus clinker rings are formed in working the rotary kiln, which sometimes block the kiln, and can only be dealt with by an experienced burner.

The ideal silica ratio in cement materials is generally agreed to be 2.5.

There are, however, some anomalies in connection with the silica ratio which go to show that at extreme limits the rules that have been given do not apply. Thus, at one works it may be found that the silica ratio is very low—viz., 1.7, and yet clinker rings are never experienced, while, on the other hand, at another works the silica ratio is very high, viz.—over 3.0, and there is little trouble with either excessive wear on brickwork or retarded hardening of resultant cement. The raw materials to be

used, therefore, and the results to be obtained therefrom are more or less a subject for local investigation.

Hydraulic Modulus.—In addition to the definitions of Portland cement which have already been cited, another but more restricted specification of cement is that contained in the following definition, viz.:—

Portland cement is a product formed by burning, at least to incipient fusion, an intimate mixture of finely ground limestone (or carbonate of lime) and clay as essential ingredients, and grinding the product to the fineness of flour, in which the hydraulic modulus lies between 1.8 and 2.2. The product shall, when made into a paste, either alone or with sand, form in a short time a hard mass, and shall show either in water or air a gradually increasing strength, and in whatever form used shall have a constant volume.

The hydraulic modulus of a cement, it should be added, is a formula which expresses the relation between the basic constituents of Portland cement, such as lime and magnesia, and the acid constituents—iron oxide, silica and alumina—by means of which a correct mixture might be calculated from the analysis of the raw materials.

The hydraulic modulus is practically the reverse of the hydraulic index equation, and is found as follows:—

$$\frac{\%\ \text{Lime}}{\%\ \text{Silica} + \%\ \text{Alumina} + \%\ \text{Iron Oxide}} = \text{Hydraulic Modulus (1.8 to 2.2)}$$

It is held that a modulus of less than 2.0 gives rise to a "dusting" of the clinker during manufacture, caused by unstable compounds, while one of over 2.4 causes expansion and cracking. A modulus of 2.2 for cement shows a high-grade product.

The **Hydraulic Index** of Portland cement is found in the ratio between the silica and alumina, the acid elements, and the lime and magnesia, the basic elements, viz.:—

$$\frac{(\text{Silica} + \text{Alumina}) \times 100}{\text{Lime} + \text{Magnesia}} = \text{Hydraulic Index (42 to 48)}$$

Commercial cements generally show an index of 42 to 48, but the French Standard Specification limits the lower index to 44.

The properties of cement are constantly undergoing changes from the time the material is ground into the manufacturer's stores until made into a mortar and incorporated into the structure; indeed, these changes continue indefinitely, and present knowledge of the internal influences affecting them is little.

Although these varying definitions indicate that no clear or concise description of Portland cement can be given, it is manifestly improper to specify the raw materials seeing that various waste products have for years been successfully used in the manufacture.

The term "artificial" Portland cement is often used in contra-distinction to the "natural" cements now commonly employed and sold in America and Belgium, and describes a carefully manufactured product obtained from the preliminary mechanical combination of carbonate of lime with silica and alumina; as outlined in the foregoing definitions, from which it will be gathered that the raw material mixing process ensures accurate and uniform results in the finished cement.

Natural Portland Cement is manufactured from mineral deposits, varying as to their chemical composition in the approximate percentages needed for making a cement. The natural deposit is burned as quarried, without any intimate mixing of the raw materials, and unless the manufacture is carefully checked a poor quality product generally results owing to its varying composition.

The composition of Portland cement is generally found to be within the following limits:—

	Minimum		Maximum
Silica	18	to	25 per cent.
Alumina	3	,,	10 ,,
Oxide of iron	2	,,	5 ,,
Lime	58	,,	66 ,,
Magnesia	.5	,,	2 ,,
Sulphuric anhydride ...	.5	,,	2.5 ,,
Water and carbonic anhydride	1	,,	3 ,,

It should not, however, be assumed from the above

limits that a cement is satisfactory in quality if coming anywhere within the range of this analysis, nor that the composition of cement is variable and uncertain.

Different raw materials vary in analysis and composition, and the resulting product, therefore, varies in its proportions in comparing one sample with another, but from whatever materials manufactured, and by whichever of the numerous processes in use, the analysis of a good quality Portland cement will generally be found to come within the limits given above.

The following table, showing the analyses of various Portland cements manufactured in this country and elsewhere, will explain what analyses of cement are met with in practice :—

ANALYSES OF VARIOUS PORTLAND CEMENTS

	Silica SiO_2	Alumina Al_2O_3	Oxide of Iron Fe_2O_3	Lime CaO	Magnesia MgO	Sulphuric Anhydride SO_3	Insoluble Alkalies Loss, &c.
Cambridge	21.04	5.12	4.50	63.04	.99	1.81	3.20
Thames ...	22.62	5.85	4.45	61.36	.99	0.97	3.75
Medway ...	22.10	7.45	2.57	63.90	1.11	0.77	2.10
Hull ...	22.22	7.26	3.97	62.53	1.34	1.86	0.82
Wales ...	20.12	6.71	4.03	60.99	2.29	1.89	3.97
Rugby ...	21.56	6.36	4.83	62.5	2.36	1.58	0.81
Hartlepool	23.3	7.85	4.65	61.9	0.90	1.43	1.97
French ..	24.92	4.80	3.28	65.32	0.95	0.48	.25
German ..	23.36	5.03	2.19	65.50	1.67	.35	1.90
American	21.66	6.13	4.47	62.20	2.58	1.60	1.36
Belgian ... (Natural)	22.14	7.85	3.40	58.76	1.53	1.02	8.30

Notwithstanding investigations by noted scientists, the chemistry, or rather the **constitution** of Portland cement is a subject not thoroughly understood. The two chief methods which have been successfully employed up to the present time in investigating the chemistry of cement are the **synthetical** and the **microscopical.** All **analytical** methods have failed, as they have failed when applied to the determination of the proximate composition of natural rocks. By long experience in its manufacture, however,

we are able to deduce from the many results of analyses the safe limits of the essential ingredients, but the manner in which these simple ingredients are bound together and the changes they undergo during the setting of cement are not clear.

The chemical constituents of Portland cement have therefore long engaged the study of experts, with the result that many different formulæ have from time to time been put forward as representing a standard composition, only to be contradicted and replaced by others offered by scientists investigating further samples of the material.

For instance, **Le Chatelier,** the eminent French chemist, years ago concluded, after a long study of the composition of cements, that the two important compounds existing in the clinker were a tri-calcic silicate ($3\,CaO, SiO_2$) and a tri-calcic aluminate ($3\,CaO, Al_2O_3$). Le Chatelier also held that in good cements the following **maximum and minimum formulæ** should be true, usually giving results in a good cement (1) 2.5 to 2.7, and (2) 3.5 to 1, viz. :—

$$\frac{CaO + MgO}{SiO_2 + Al_2O_3} \leq 3$$

and

$$\frac{CaO + MgO}{SiO_2 + Al_2O_3 + Fe_2O_3} \geq 3$$

Again, conclusions differing from the above have since been arrived at by **synthesis,** and it has been observed by other experts that experiments as to the constitution of cement led to the following results :— (*a*) Lime may be combined with silica in the proportion of 3 molecules to 1 and still give a product of practically constant volume of good hardening properties, though hardening very slowly. With $3\frac{1}{2}$ molecules of lime to 1 of silica the product is not sound, and cracks in water. (*b*) Lime may be combined with alumina in the proportion of 2 molecules to 1, giving a product which sets quickly, but shows constant volume and good hardening properties. With $2\frac{1}{2}$ molecules of lime to 1 of alumina the product was not sound.

The Le Chatelier formulæ, however, expressed in chemical equivalents and applied to commercial cements,

require a considerable calculation, but at the same time it is admitted that they have the merit of taking into account the difference in the combining power of silica and alumina, and in this respect are a great advance over the hydraulic index and modulus.

Other authorities suggest slightly modifying the Le Chatelier maximum formula and express it in weights instead of equivalents as follows :—

$$\text{Lime} = \text{Silica} \times 2.8 + \text{Alumina} \times 1.10.$$

This formula gives the maximum of lime which a cement **raw material** mixture can contain, and give sound cement, under ordinary conditions of burning. If we substitute 1.0 for 1.1 in the formula (which makes no practical difference in the result) we have another formula, or **lime factor** as it has been termed, thus :-

$$\frac{\text{Lime} - \text{Alumina}}{\text{Silica}} = 2.8$$

which represents the maximum of lime permissible. Raw material mixtures in which the lime *minus* alumina divided by silica, is more than 2.8 are likely to yield an unsound cement.

On the other hand raw material mixtures in which this figure is less than 2.6, generally give a fusible clinker and quick-setting cement. So, for practical purposes, it has been recommended that the adoption of 2.7 as a standard lime factor for **raw materials** should be satisfactory, and this decision is, doubtless, as theoretically accurate as the present and past experience of cement manufacture will allow of calculation.

Commercial Portland cements, it is shown, generally possess a decidedly lower lime factor than that for raw mixtures. The **lime factor in cements** (lime *minus* alumina divided by silica) is generally from 2.5 to 2.6. This is due to the change in composition of the mixture effected during burning by taking up the ash of the fuel.

From the foregoing descriptions, therefore, it will be seen that the **essential ingredients of cement** are the compounds of lime, silica and alumina, and in nearly all cases Portland cement, provided it is of good quality, has practically the same composition.

The Mineralogy of Cement.

CHAPTER III.

As the cement consumer when in doubt generally turns from the practical use to the mechanical testing of cement for a definite pronouncement upon the quality of any given product, so the cement chemist passes from the chemical examination to the mineralogical in his endeavours to understand more thoroughly the precise answer to the question so often asked: "What is Portland cement?"

If what has been published from time to time, however, is all that is known up to the present concerning the mineralogy of cement, this science has given us little information and still fails to answer the query.

It will have been seen from the foregoing chapter that the constituents of ordinary Portland cement can readily be ascertained by the usual chemical tests and methods of analysis, and from such an investigation it is also found that the composition of all good cements differs but slightly, except in so far as the percentage of the ingredients contained in the different samples varies. No chemical examination, however, enables the expert chemist or anyone else to judge whether a brand of cement in its practical sense is of a superior or an indifferent quality, since it would not be a difficult task to prepare a material containing the chemical combinations and the exact constituents of ordinary Portland cement, but which in practice would possess no cementing properties whatever. The determination of the market value of the product must still, therefore, be left to the mechanical testing, which is explained at length in the following chapters.

In spite of this position, however, the Portland cement industry cannot by any means dispense with a thorough knowledge and practice of chemistry in the manufacture, testing and uses of cement, for this knowledge not only gives valuable information upon the variations or uniformity of the finished product, but chemistry explains the peculiarities of the raw materials used in the manu-

facture, which is a highly important point requiring especial study. In the proper mixing, also, of the raw materials, chemistry comes to the manufacturer's aid and, regularly applied, keeps a resulting product uniform in quality.

As the manufacture of a faultless cement cannot be left to chance, and as absolute accuracy is, moreover, essential in the manufacture of a good quality product, so it will be seen that a comprehensive knowledge of chemistry is required in dealing with Portland cement; but whereas chemistry has long been in the service of the cement industry, mineralogy is only now gradually entering this sphere.

In the study of Portland cement, mineralogy is likely to be an excellent teacher, for it supplements the technical and chemical investigations, and, indeed, it has already made many an important disclosure. A good microscope has, therefore, become an indispensable item in the equipment of the cement-testing laboratory.

Mineralogical study has shown that all Portland cements consists of a subtle mixture of compounds allied to minerals. This fact is not disputed by experts, even though it may not yet be a subject of general knowledge.

An advanced view of the constitution of Portland cement clinker is contained in a statement made to the author in New York some years ago by Mr. Clifford Richardson, and to it are applied the same theories of solution which have proved so fertile in metallurgy.

It is well known that the mineral constituents of rocks can be identified under the **microscope** in thin sections, especially with the aid of polarised light, and these petrographic methods have been applied to sections of Portland cement clinker.

The two chief minerals which have been recognised in this way in cement clinker have received the names **Alit** and **Celit.**

Other constituents are also recognisable, but these appear to arise from non-essential constituents—such as iron, magnesia and the alkalies.

Working with perfectly pure silicates of lime and aluminium, success has been attained in forming synthetically correspondingly pure contents, consisting wholly of Alit and Celit. By varying the proportions

of the different constituents taken, it is found that **Alit** is a solid solution of tri-calcium aluminate and tri-calcium silicate, whilst **Celit** is a solid solution of di-calcic silicate and di-calcic aluminate. In the fused state these aluminates and silicates are soluble in each other in all proportions; but this is not the case in the cold, the mixture then splitting up into alit and celit, each of which has the properties of a true cement. In order that both constituents may be present, the percentage composition of the cement must be between the following limits :—

Silica	...	...	18.5 to 23.2
Alumina	...	...	6.1 to 11.9
Lime	...	...	63.1 to 68.1

In commercial work, cements do not reach the higher limits of lime, as it is impossible to obtain industrially the high temperature then needed in firing them, but this can be satisfactorily carried out in the laboratory. In practice, of course, cements are not fused, but merely fritted in the process of manufacture, and the formation of these solid solutions by fritting the materials together at a temperature much below their point of fusion is attributed to that process of diffusion of one solid into another, which was shown to occur in the case of metals. The higher the temperature, the more rapid this process of diffusion. The time requisite will also depend upon the area of the surfaces between which it can take place. Hence, the finer the grinding of the raw materials the more quickly and easily is the diffusion operation accomplished. A danger to be guarded against is, however, the use of a very high temperature for fritting, followed by a very rapid cooling of the burnt material. The calcium aluminate is much more soluble in the silicate at high temperatures. If the cooling is too quick, a supersaturated solid solution is obtained, which is, of course, in an unstable condition.

The change to the stable forms is accomplished by a change of volume, and, therefore, destructive of the cohesion of the clinker.

Another view upon the technology of cements has been expressed to the author by Messrs. Day and Shepherd, of the Geophysical Laboratory of the Carnegie Institution of

New York, who have recently shown that tri-calcium silicate does not exist in the two-component series.

It is known that scientists have been labouring for years to discover the constitution of Portland cement, and mineralogy now teaches us that this search if conducted alone upon the lines of chemical research is a useless endeavour and a waste of time.

It seems clear, for instance, that Portland cement has no fixed constitution, because it is not a homogeneous body. It is a mineralogical mixture; a compound of several bodies, which are Portland cement bodies or Portland cement minerals. This discovery is very important and very significant, because it brings us considerably nearer the answer to the question which was asked in the opening to this chapter, and much nearer a final solution.

Cement experts are agreed that there are a number of Portland cement minerals. So far as is known at the present time, the number is stated to be five, but in time perhaps others will be discovered. The study of these materials is complicated, and far from easy, for the five Portland cement minerals do not by any means occur uniformly in each brand of Portland cement, neither in the same quantities nor even in the same manner, and sometimes one mineral or more is missing altogether. In the opinion of some experts, all the Portland cement materials have, in spite of their diversity, the same origin, otherwise they would not be what they are. They belong to the genus cement, burnt out of lime and clay, and freed from all carbonic acid. The Portland cement minerals, so far as is known to-day, consist of four crystalline and one amorphous mineral. Each of these has its own constitution, but the actuality of none of them has yet been discovered with any certainty.

The names of the crystalline Portland cement minerals are given as Alit, Belit, Celit and Felit. The amorphous mineral has been termed vitreous or "glassy" residue.

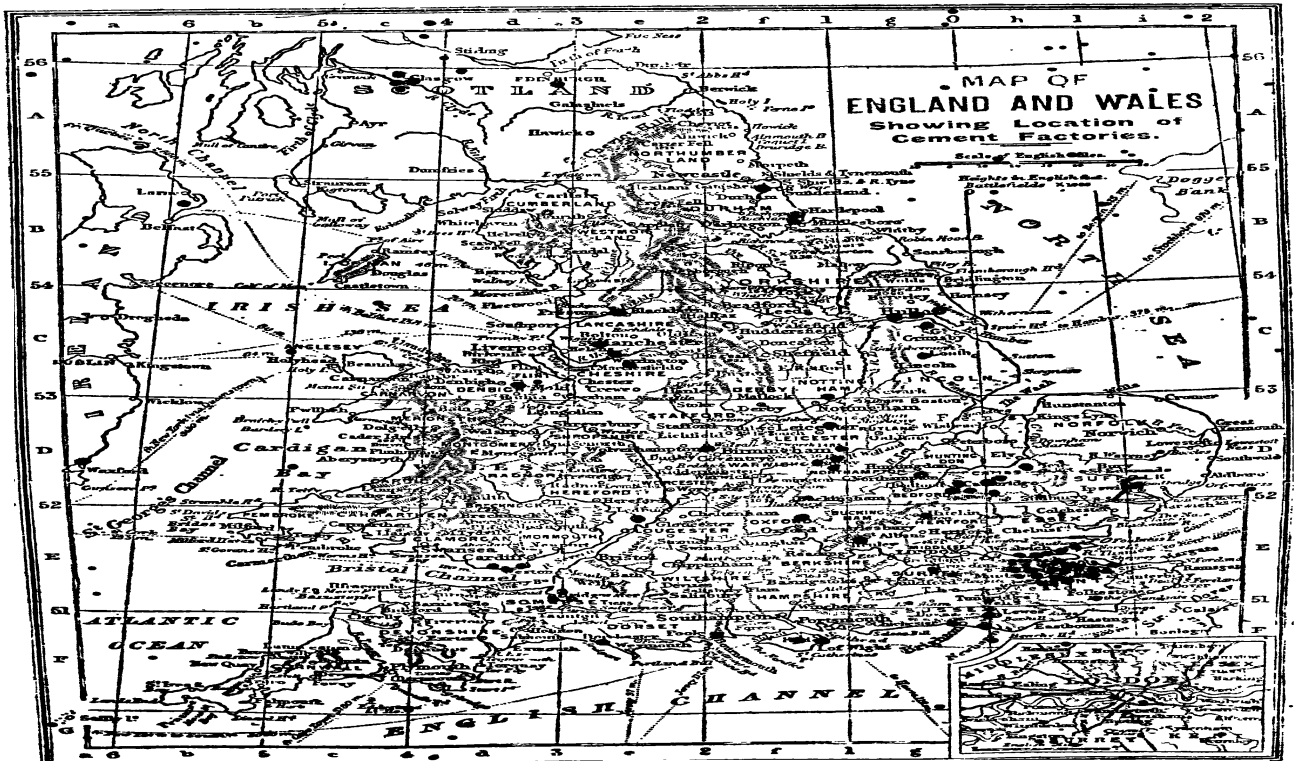
To the expert following up the mineralogy of Portland cement the two most interesting and important of these five minerals are the alit and the glassy residue. A noteworthy body also is the felit. From such investigations as have already been made, it is not possible to say much about the actions and functions of belit and celit, for at the present they appear rather as a kind of ballast in the

An interesting view concerning the mineralogy of cement and its connection with the disintegration of cement clinker when burned under very high temperatures may be quoted:—When clinker is burned at a temperature higher than is necessary for its crystallisation, it contains much of the mineral called glassy residue. Such clinker when allowed to cool off gradually begins to form crystals, and so to lose some of its glassy character. This process causes the mass to disintegrate. If, however, such a clinker is suddenly chilled with cool water, then the Portland cement glassy residue is quickly changed from its plastic condition into a hard and rigid one, and the crystallisation is made impossible. The clinker thus not only remains permanently intact in form, but it also retains the great hydraulic qualities of the glassy residue mineral in Portland cement, and thus preserves for itself its own hydraulic value.

It is clearly seen under a powerful microscope that felit is a mineral having a well-defined double plane of fracture, plainly striped in parallel, while alit is colourless, granular or flaked, and with weakly-defined double planes of fracture.

The last of the minerals—namely, the Portland cement "glass"—is believed to occupy a subordinate position in the mineralogy of cement, but experts are not agreed upon this point. According to some experience, it is found that this glassy residue may, under certain circumstances, occupy as important a *rôle* as alit, and it has been held that under correct conditions it possesses exactly the same energy for hardening.

Portland cement "glass" examined under a microscope appears as a colourless glass, but in isolated cases it appears slightly yellow. When it is ground fine, made up with water and compressed, it dissolves exceedingly slowly at ordinary temperatures. It dissolves more quickly if hot-water is used, and extraordinarily quick in an alkaline solution.


MAP OF
ENGLAND AND WALES
Showing Location of
Cement Factories.
SCOTLAND
IRISH SEA
NORTH SEA
ATLANTIC OCEAN
ENGLISH CHANNEL
Bristol Channel
Cardigan Bay
St. George's Channel

Raw Materials.

CHAPTER IV.

PORTLAND cement can be produced from any raw materials whose constituents are capable of yielding by calcination the silicates and aluminates of lime which form its chief components.

The **necessary raw materials** for the manufacture are lime (usually in the form of carbonate of lime, such as chalk and limestone), silica and alumina, commonly found in their combined form as clay or shale. Lime, silica and alumina are also obtained for cement manufacture in indefinite proportions from calcareous tufa, slags and alkali waste, although such materials are only employed to-day to a minor extent.

In the chalk-marl and lias limestone deposits over the country the necessary ingredients are found in approximately correct proportions for cement manufacture in the one deposit, naturally mixed, but rarely, if ever, in the exact proportions required.

In order to be suitable for the manufacture of cement the raw materials must be of correct chemical composition, which implies that they should contain as small a percentage as possible of **magnesium carbonate**—no more than 1 per cent., which is the maximum permissible. Free or **insoluble silica** in the form of chert, flint, or quartz should either be entirely absent or present only in small quantities, and **sulphate of lime** should not exceed 2.5 per cent.

On the other hand, **iron oxide** and **alumina** are necessary or desirable for fluxing during calcination, but an excess of these substances is objectionable.

It will thus be seen that with chalk and limestone, the other necessary materials, namely, river mud, shale and clay used for forming the respective admixtures with the chalky materials should be mainly silicious, but as free from sand as possible. Highly silicious clays, containing silica up to 75 per cent., give mixtures which stand the great heat of the kiln without fusing, and produce a clinker

which is comparatively easy to grind, and which generally yields a slow-setting cement, showing a steady increase in tensile strength over long periods.

The two materials for the necessary chemical combination indispensable for the manufacture of cement are not always found upon the same site. Some works, however, are favoured with these conditions, and it is only necessary to examine the localities of the most successful works in the country to see how invariably it is the case that, according to the facility with which they command the raw materials and their proximity to the market, their successes have been assured.

From considerations of economy in the excavation, crushing and reduction of the raw materials it is desirable that they should be as soft and as dry as possible, but this is not essential as far as efficiency is concerned.

A Portland cement plant running on dry materials, such as a mixture of limestone and shale, will use approximately 16,000 tons of raw material per year for a weekly output of 200 tons of cement. Of this, about 12,000 tons are limestone and 4,000 tons shale. Assuming that the limestone weighs 160 lbs. per cubic foot, which is a fair weight, each 200 tons per week output will require about 168,000 cubic feet of limestone per year. As the shale may be assumed to contain some considerable amount of water a cubic foot will probably contain not over 125 lbs. of dry material, so that each 200 tons cement output per week will also require 72,000 cubic feet of shale. If calculating, therefore, for the location of a plant with a 20 years' supply of raw materials, there must be in sight for every 200 tons output of cement per week at least 3,360,000 cubic feet of limestone and 1,440,000 cubic feet of shale.

The raw material deposits for the manufacture of Portland cement are widely disseminated, but the suitability of a certain **locality** depends upon the manner of the occurrence of these requisites and the location of the deposit with respect to the cement consuming market and the fuel supplies.

Thus, the building of factories will be centred at a point where the raw materials are to be found in large quantities, and near together, where their physical character is such as to render them easy of getting and clearing, and of

comminution and mixing, where coal or other desirable fuel may be had at low prices, and where the market is not too far removed.

As has been pointed out, from a geological formation of uniform composition containing exactly the right amount of naturally mixed lime, silica and alumina, Portland cement could be made by the cheap and simple method of merely burning lumps of the material as obtained from the quarry, and grinding the resultant clinker. Such a material has not been discovered in quantity in this country, and it is perhaps hardly likely that it will be, for a variation of even one per cent. of the proportion of carbonate of lime in the raw material for the manufacture, to say nothing of the irregular composition of its other ingredients, is sufficient to destroy at once the reliable quality of the resultant cement.

In manufacturing Portland cement, therefore, carbonate of lime and clay are first efficiently and accurately amalgamated in certain fixed proportions, either by the addition of water to the raw materials, or by the fine grinding and mixing of the same in their dry state.

With either a wet or dry process of blending the raw materials to a fixed percentage of carbonate of lime, the fineness of the minute particles must be such as to allow of the exact chemical change taking place in the burning or calcining operation, and if this preliminary process of mixing the raw materials be not carried out with sufficient thoroughness, failure is bound to be the result, and will at once make itself evident when the material is further dealt with.

The hardness of the raw materials treated in the manufacture of Portland cement is of great importance in determining their practical value, since the constituents must be ground to great fineness before mixing. Pure limestones require pulverising to an impalpable flour, almost all passing a 180 by 180 mesh sieve, and are, therefore, costly to treat; whereas other limestones or chalk-marl containing a considerable proportion of clay are generally much softer, and the mixing being already done in part by nature, a better burning combination and finished cement often result.

The **analysis** of a prepared **mixture** of raw materials

for the manufacture of cement may be approximately as follows :—

	Per Cent.
Carbonate of Lime	76.35
Silica	14.46
Alumina	5.33
Iron Oxide	1.69
Magnesia	0.04
Sulphuric Anhydride	0.31
Potash, Soda, &c.	1.82
	100.00

In or about the year 1826 chalk in the neighbourhood of the River **Medway** was first used with the river clay or mud for experiments in the manufacture of cement, and a few years later the first cement works in this district were established at Northfleet, in Kent.

At this time the use of **Roman cement** which is manufactured from argillaceous limestone having a large proportion of clay was considerable, and although the then new cement was found to be a more reliable material there was naturally some opposition to the introduction of the product.

At the present day, however, the manufacture of Portland cement is forming the staple industry in this neighbourhood, and indeed it is not only in this district that large and important factories have sprung up, but successful works are now situated in many other parts of the country.

The chalk formation on the banks of the rivers **Thames and Medway,** and the deposits of mud in the estuaries and creeks have, however, furnished a suitable supply of raw materials in an acceptable form for the manufacture of cement. **Analyses** of these materials are given below :—

	Chalk.	Mud.
Silica	1.52	65.02
Alumina	.85	19.74
Iron Oxide		7.22
Carbonate of Lime	96.45	1.52
Loss, &c. (and organic matter) ...	1.18	6.50
	100.00	100.00

For many years the only materials used in the manufacture of cement were chalk and clay, the **white chalk,**



CHALK QUARRIES ON THE MEDWAY.

which contained as quarried about 18 per cent. of water, being exclusively used on the Thames; and on the Medway, the **grey or lower chalk** found underlying the white chalk in the hills bounding the valley of the river.

These chalk deposits have a depth of 100 to 300 feet, and the excavating is generally carried on by automatic plant, the material being then loaded into trucks for conveyance to a distance by locomotive direct into the factories.

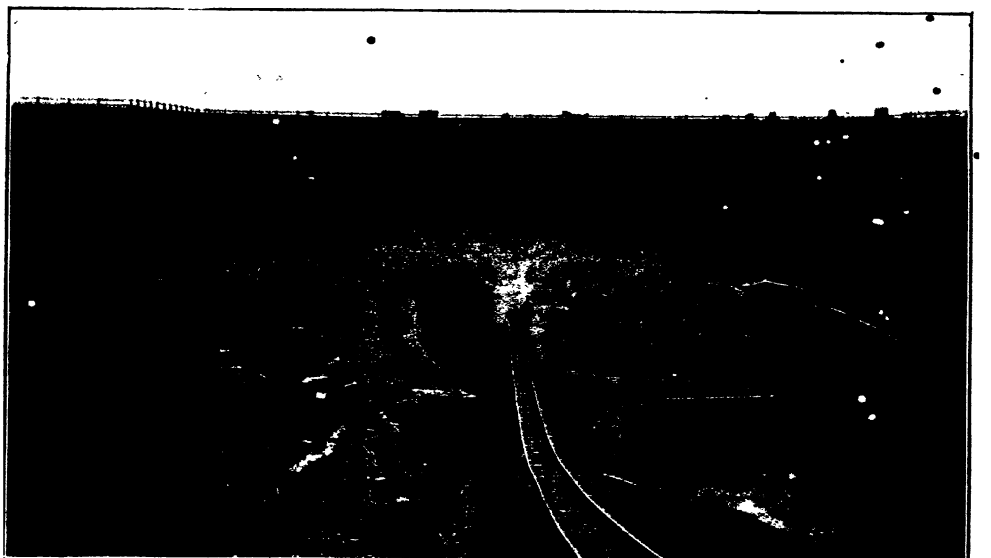
The greater part of the **clay** used by the Thames and Medway manufacturers is the alluvial mud referred to and is found in the estuary of the rivers; although on the Thames the inner part of the "saltings," as they are termed, is excavated from the banks of the river and used in the manufacture.

The **chalk marl around Cambridge** is a deposit of naturally mixed calcareous and argillaceous material found at the base of the local chalk. It differs from the main mass of the chalk above it in the quantity of clay it contains. The Cambridge greensand underlying the deposit of chalk marl is a chalky bed a few inches thick, with phosphatic nodules and green glauconitic grains. This deposit marks a more or less stationary period in respect of sedimentation before the deposition of more calcareous debris set in.

It is a curious fact, and one worthy of note, that the chalk marl of Cambridgeshire, though the relative quantities of chalk may vary from yard to yard in depth when testing on the face of a quarry, contains in the aggregate about the same proportion of calcareous and clayey material as is obtained artificially by the mixture of chalk with alluvial mud in the Medway Valley. Therefore, along the outcrop of this chalk marl stratum cement works have of recent years proved the value of the Cambridge deposit from a cement-making point of view.

The deposit is, in the most favourable conditions, found about 2 feet below the surface, and runs to a depth of from 30 to 60 feet.

The chalk contained in the material, being in a measure the most valuable and most important ingredient in the making of Portland cement, is shown from various analyses to average 70 to 80 per cent., and the remaining constituents are exactly the amounts of silica, alumina, and



QUARRY SHEWING CHALK MARLS OF CAMBRIDGE DISTRICT.

oxide of iron required in the manufacture. It is only safe to treat these marls under a scientific cement-making process, and the manufacture is arranged so that the "lower chalked" marl is intimately blended with the material containing an excess of lime-carbonate, so as to produce a working average of, say, 76 per cent. The resultant mixture is then ready for the further operation of burning.

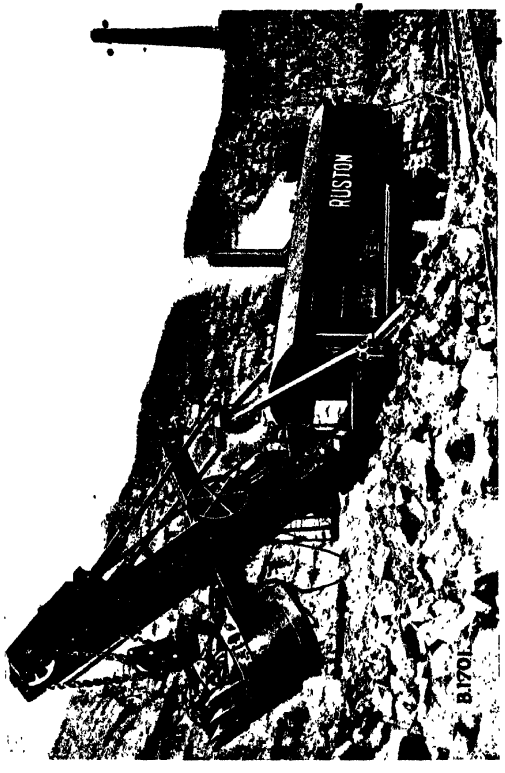
In the early days of cement-making, some of the marl deposits of the Cambridgeshire district, which very nearly approached the desired combination of chalk and clay, were used for manufacture without any mechanical mixing. In such instances the manufacture would be carried on by burning the calcareous deposit just as it is quarried, and grinding the resultant "clinker" (a large proportion of which would be dust) for Portland cement. The unreliable quality of a material so produced is demonstrated by the crudest tests; and, from a scientific as well as from a commercial point of view, where the deposit varies in its composition, as the analyses of chalk marls generally show that it does, such a process of manufacturing Portland cement—a chemical product—cannot be too strongly condemned.

Given a scientifically mixed and corrected raw material from the marl deposit, which is really an intimately blended chalk and clay mixture, a cement can be produced yielding excellent results, which has been shown to be equal to the Thames and Medway cements prepared from the wholly artificial and mechanical mixtures referred to above. The following is an **analysis of mixed raw materials** prepared for the manufacture of cement from the Cambridge deposit :—

Silica	15.72 per cent.
Alumina	4.20 ,,
Iron Oxide	1.89 ,,
Carbonate of Lime	76.60 ,,
Carbonate of Magnesia ...	1.31 ,,
Undetermined	.28 ,,
	100.00 per cent.

In the cement-making districts of **Rugby and South Wales**, the well-known Lias formation of limestone and

shale is successfully treated, and the thin layers of these materials, with their considerable variation in composition, must be very accurately and efficiently dealt with. The deposit may be found to an appreciable extent in other



STEAM SHOVEL EXCAVATING LIAS LIMESTONE.

parts of England, but the preponderance of shale in proportion to the limestone (which is generally found to contain only 78 to 85 per cent. of carbonate of lime) renders the cost of working excessive, because of the amount of

material not required in the manufacture. The raw materials here are sometimes ground in their dry state for the preliminary operation of mixing to the necessary percentage of lime-carbonate, and when carefully dealt with Portland cement of excellent quality is manufactured.

The **Lias formation** consists of layers of impure limestone of varying thickness, ranging from two inches to two feet, and parted by seams of shale and clay—the total thickness of shale beds considerably exceeding as a rule that of the limestone.

Analyses of the Lias **limestone** and **shale** are here given :—

	Limestone.	Shale.
Silica	7.81	30.05
Iron Oxide	1.90	10.70
Alumina	2.59	3.35
Carbonate of Lime	81.37	46.88
Carbonate of Magnesia	1.72	3.68
Alkalies and loss	1.61	5.34
	100.00	100.00

On the **Tyne, Wear** and **Tees** and in the North of England generally, Portland cement was manufactured from chalk imported from the Thames, Medway or Sussex districts, and mixed with a local clay, found on the site of the works, which contains less organic matter than that used in the South. From this mixture a good quality cement is made.

In **Hull** the chalk deposits on the banks of the Humber are used with local clays. In **Northamptonshire** the oolite limestone and lias clay, which are sometimes superimposed one above the other, are used as raw materials.

In the **United States,** most of the cement produced is from the Lehigh Valley argillaceous limestone, which contains rather more clay than is required for a correct mixture. To this a small amount of pure limestone, usually 10 to 20 per cent., is added to bring the mixture up to the

necessary percentage of calcium carbonate required in the manufacture.

The chemical composition of certain typical forms of raw



Heavy Steam Shovel Excavating Hard Chalk.

material used in the manufacture of cement in the States is here given :—

Calcium Carbonate	Magnesium Carbonate	Calcium Sulphate	Silica	Alumina	Iron Oxide, &c.
88.16	1.78	-	8.20	1.00	0.86
70.10	3.96	–	15.05	9.02	1.87
68 81	4.28	--	16.32	7.07	3.52
91.77	0.53	3.19	0.22	1.22	3.07
88.49	2.71	1.58	1.78	0.91	4.53

It will thus be seen that given chalk and clay, or a carbonate of lime with silica and alumina in any other form, it is of first importance that these materials shall be treated with a full knowledge of the all-important chemical and mechanical operations of combining them, which alone can secure the manufacture of a reliable product.

Raw Materials—Mixing.

CHAPTER V.

THE preliminary mechanical blending of the raw materials for the manufacture of Portland cement is an operation requiring the utmost technical skill; for the thoroughness of the mixing in the amalgamation of the raw materials primarily determines the quality of the resulting cement; and if the manufacturer is to turn out a product of reliable quality, the supervision of this branch of the manufacture is of paramount importance.

The time has gone by, therefore, when this responsible duty was left to the ordinary workman to perform by rule-of-thumb methods, and the mixing of the raw materials in the most successful cement works is now carried out under the closest supervision of the works' chemists. The manufacturer thus well knows that the finished product is not exposed to the continual dangers and failures in test which were so common in the earlier stages of the industry.

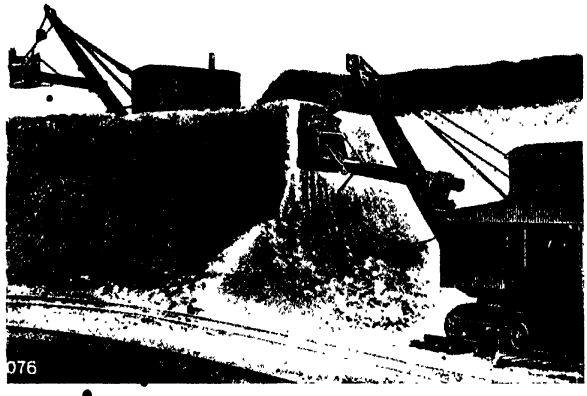
The primary conditions of any methods of Portland cement manufacture are that the raw materials shall be correctly proportioned, very finely comminuted and thoroughly mixed.

How these conditions can best be fulfilled depends chiefly upon the character of the raw materials; so that the details of the methods adopted by manufacturers generally vary with the raw materials used.

For example, in the Thames and Medway districts, the soft chalk, together with the river mud, is advantageously mixed by "washing" the two materials together, and where chalk-marl is used, or limestone and shale, these sometimes are reduced by both wet and dry grinding. With either process the fineness of the particles must be such that the exact chemical change is allowed to take place in the next operation of the manufacture (burning or calcining), and if this preliminary process of mixing the raw materials be not scientifically carried out, failure is bound to result when the mixture is subjected to the heat necessary for calcination.

The first process in the manufacture of cement is generally the uncovering of the deposits and the quarrying or mining of the raw materials, in which operations the most approved plant is now employed to save labour.

The general use of mechanical diggers is superseding methods of hand quarrying in chalk quarries where the "milling" of chalk has prevailed for many years. The latter system of barring down the chalk in steps by hand labour involves the loosening of the material, which then falls direct into trucks at the bottom of a semi-circular



EXCAVATORS TOPSOILING AND SOFT CHALK QUARRYING.

shaped face for the conveyance of the raw materials to the factory.

In the case of lias limestone and shale, the same mechanical digging plant is employed, after drilling, blasting and shattering of the deposit has taken place, but in this instance the addition of further plant is necessary in the shape of a "rumbler," or revolving circular framework, for shaking out the preponderating shale which exists with such materials. Clay, on the other hand, can either be won by a grab or by the usual excavating methods employed in dealing with the identical material locally obtained.

After the quarrying of the raw materials and their conveyance to the factory, there are two principal methods of mixing them, namely :—

(1) The "Wet process," by which the materials are mixed with water, and reduced to an extremely fine state of division by grinding machinery.

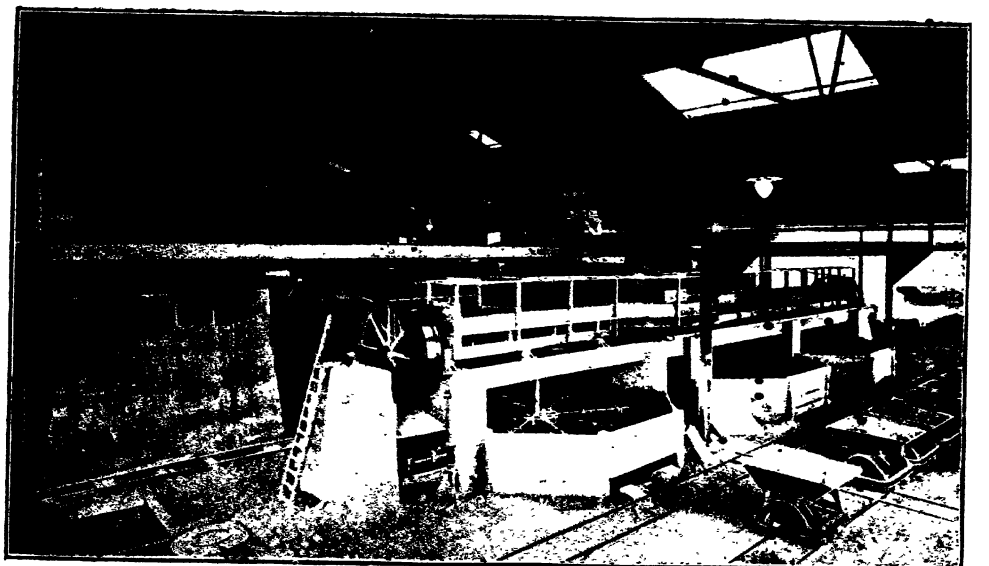
(2) The "Dry process," in which the perfectly dried materials are ground together to an impalpable powder or "flour," and subsequently mixed to correct chemical proportions.

The wet process of mixing is particularly adapted to such materials as are easily disintegrated by the addition of water, and this method of mixing is employed to a considerable extent by the manufacturers of cement on the Thames and Medway.

The original wet process—now obsolete—consisted of tipping chalk and clay in weighed quantities into a wash-mill and adding a large proportion of water to the mixture. The wash-mill usually consisted of a brick pit about 15 feet in diameter and 5 feet deep, in which revolved a series of harrows fitted with "tines" or teeth for breaking up and mixing the two ingredients in their wet state. It was fitted with a circular framework, revolving on a central pier, to which the harrows were suspended by chains, the machine being driven by a crown wheel, which travelled the heavy harrows round the wash-mill and through the material at the same time, at a speed of about 15 revolutions per minute, thus agitating the mass and disintegrating and mixing the raw materials, to which water was added in such quantities as to bring the mixture to about the consistency of milk. In this condition it is termed "slip" or "slurry."

It is, of course, essential that extreme care should be exercised in adding the correct proportions of the chalk and clay, or whatever other raw materials may be used in the wet process, so that the resultant mixture may be relied upon to contain the exact chemical constituents necessary for the manufacture of a thoroughly sound cement; for if the raw materials are not mixed in this proper proportion, nothing can be done later to correct it, and the result will be an inferior product.

When the liquid mixture of chalk and clay was sufficiently fine to pass the grating with which a portion of



WASH MILL PLANT—WET PROCESS.

the periphery of the wash-mill was fitted, it flowed into settling "backs," where some of the excess of water added was removed by decantation, and when the slurry became sufficiently solid to be handled with a shovel, it was conveyed to kilns for burning.

Manufacture by this wet process was, however, attended by certain drawbacks, one of which was the lengthy period taken up in drying—a period ranging from six to twelve weeks. Another, and one which was far more serious, was a liability to inequality in the mixture. This was caused as follows:—When the slurry has been deposited in the "backs" the particles of chalk settle more quickly than the particles of clay with the result that the contents of the reservoirs consist of layers of over-chalked material, intermixed with layers of over-clayed material, and thus the certainty of obtaining the uniformity of mixture necessary for the best results is rendered impossible.

These experiments led to the adoption of the semi-wet process of raw material mixing. This was the system originally patented by the late Mr. W. Goreham for washing the raw materials with a minimum quantity of water, whereby the raw material mixture then used in the manufacture was changed from a thin chalky consistency to one of a much thicker mixture or one of the consistency of mud. With the substantial change in the manufacture brought about by this improvement in the raw mixing process there appears to have been a general alteration of the manufacturing plant. It was then maintained that by materially reducing the amount of water, and grinding the mixture by mill-stones, such as were then in use for grinding the finished cement, the grinding and mixing of the raw materials were rendered more thorough and complete. The original form of grinding plant for what was then termed the "semi-wet" method consisted of three pairs of mill-stones, placed in such a way that the thick slurry after grinding from the first pair ran to the second and through to the third.

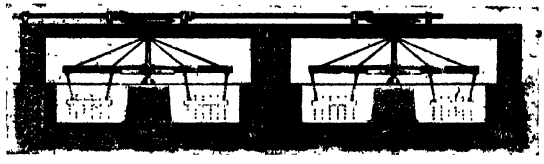
This repeated grinding was found, however, to be unnecessary, and the grinding mills were then so arranged that the thick chalky mixture ran only once through, and ultimately this grinding of the wet slurry gave way to a method merely of separation.

The original patent granted for the manufacture of cement by the semi-wet method is dated July 12th, 1870, which was of course before the introduction of the Rotary Kiln. The patent runs :—

" Portland cement is made from chalk and clay.

" There are at present two different methods of manufacturing Portland cement.

" According to the **wet method** commonly practised in England the chalk and clay are first ' washed ' in wash-mills with harrow tines, and are so mixed with about five times their weight of water. They are thus thoroughly disintegrated and mixed together, and the mixture flows out of the wash-mills as a liquid of about the consistency of milk.



Section of Wash Mills Employed in the Wet Process.

" The further process of manufacture is the separation of the water which has thus been mixed with the chalk and clay in order that the ' slip,' as the mixture is called, may be sufficiently dried for burning in kilns.

" The liquid mentioned is therefore run into reservoirs or ' backs,' in which the chalk and clay gradually sink to the bottom, and the water above it for the most part removed partly by drainage and partly by evaporation. This process occupies from six to twelve weeks, according to circumstances.

" The chalk and clay are then dug out of the ' backs ' in the state of mud, more or less thick, and the ' slip,' after having been further completely dried by artificial means, is then burnt in kilns and ground.

" According to the **dry method** as frequently practised, the chalk and clay are each ground separately, having first been artificially dried to facilitate the grinding. They are then mixed, and the mixture thus resulting is made into

bricks or lumps by the aid of just sufficient water to bind it together. The bricks or lumps are then burnt in kilns with or without being previously dried on stoves or otherwise.

"Now, according to my invention (a semi-wet method) the chalk and clay are roughly mixed together in a wash-mill with a small quantity of water, only, say, one-third



PORTLAND CEMENT FACTORY IN 1856.

of the weight of the whole. The product resulting from this operation is not a liquid but a mud with coarse particles of undisintegrated chalk and clay interspersed throughout it.

"I then pass this mud through a pair of mill-stones, such as are now used for grinding cement after it comes from the kilns, and thereby thoroughly comminute and mix the whole. The mud is then immediately transferred

to the drying stoves or chambers without being poured into the reservoirs or 'backs' and the dried slip is then burnt in kilns.

"A portion of the fuel for burning the cement may be mixed with the chalk and clay, as is described in the specification of a patent granted to me and another and dated the fourth day of February, one thousand eight hundred and seventy, No. 99. In such case the fuel is added to the other materials in the wash-mill.

"I avoid by my process the time, space and expense involved in driving off the excessive quantity of water with which the chalk and clay are mixed according to the wet way of manufacture, inasmuch as I succeed in effecting the perfect disintegration and mixing of the materials with a small quantity of water only and in thus obtaining a product which can at once be dried by artificial means without the aid of drainage or evaporation in backs.

"I also avoid the expense incurred in the so-called dry process by drying and grinding the chalk and clay separately, and in afterwards mixing them with water, and I also obtain finer comminution and more uniform admixture of the materials than it is believed can be obtained by the dry method.

"Although I have adopted by preference the above method of partly mixing and disintegrating the chalk and clay in a wash-mill with harrow tines and afterwards of completing the process by further grinding the mud between mill-stones—this method may be varied, but I claim as my improvements in the manufacture of Portland cement, disintegrating and mixing the chalk and clay by grinding or crushing when wet, but without excess of water, and then at once drying and burning the mud thus obtained without the separation of water in settling backs."

It will thus be seen that the difference between the original wet process and the semi-wet process was, that in the former the whole of the grinding and mixing was done by the wash-mill with very large additions of water, while in the semi-wet process a much lower percentage of water is used, the wash-mill only preparing the work of grinding and mixing; the final reduction and mixing being accomplished by means of mill-stones or other convenient grinding machinery.

The semi-wet process was found to be a considerable improvement upon the older methods, and has, with modifications, since been adopted by the majority of manufacturers throughout the country making cement by the wet process.

Under the newer method the necessity for the large amount of water used in the original wet process—and which must all be dried off before the material can be burned—is obviated, the slurry being carried on to the further machinery of reduction or separation, where the operation of amalgamation is completed.

It was found, however, as time went on that the wet grinding of the raw materials served a double purpose: it not only reduced the raw materials to such a size that the necessary intimate mixture might take place, but it also assisted the actual mixing, which it rendered more uniform, and consequently improved the quality of the finished cement.

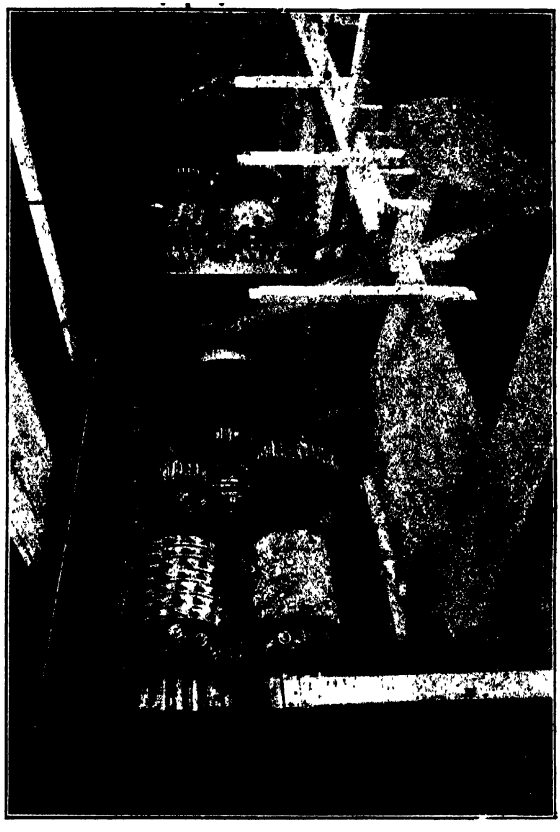
The **wet process** as now adopted on the Thames and Medway, consists in tipping the weighed raw materials into a wash-mill similar in construction to that previously described, but having a coarse grating instead of the fine screens before mentioned. The partially disintegrated chalk and clay with water amounting to about 42 per cent. of the whole, passes through this grating into another wash-mill, known as a screening mill, which completes the disintegration, and which is fitted with a fine screen, so that no particles which will not pass a sieve of about 40 meshes per linear inch can escape.

In some cases a series of three wash-mills is required for the complete disintegration and screening of the chalk and clay, while in other cases, where the chalk is soft, it is possible to perform the whole operation in one wash-mill.

Again, in some cases the slurry is passed through a tube-mill after leaving the wash-mills in order to perfect the grinding. Such a tube-mill consists essentially of a long revolving drum made of steel and protected against wear and tear by renewable cast-iron plates. The drum is about half filled with rounded flint pebbles, and is fitted with one or more manholes for the purpose of access.

A modern modification of the wet process is sometimes

referred to as the "thick slurry" process, which is an adaptation of the wet process for use with hard raw



Crushing Raw Materials—Dry Process.

materials, such as limestone and shale. In the thick slurry process the weighed raw materials are crushed and conveyed to a ball-mill—a large steel drum containing heavy steel balls—which pulverises the material to be

ground by impact. Water is also introduced into this wet ball-mill, and the result is a partly ground slurry, which passes to a tube-mill for final reduction.

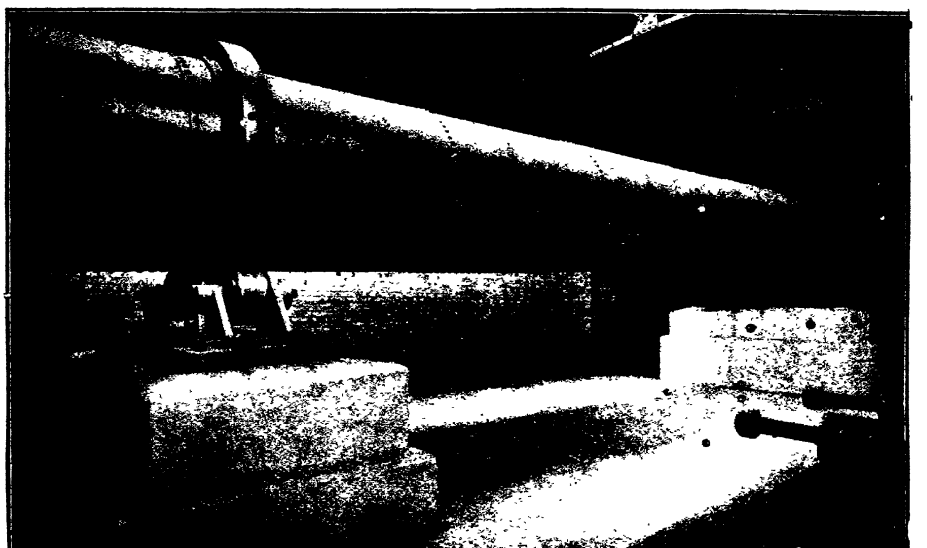
In the **dry process** method of dealing with the raw materials for cement manufacture the calcareous and argillaceous materials, of whatever substances they may be composed, have first to be dried after passing the preliminary crushing machinery.

The dryers used in the manufacture of cement have in the past taken the shape of expensively constructed kilns or heated floors, with their attendant heavy working charges, but with the more general adoption of the dry process of manufacture in other countries little time was lost in inventing a less costly and more efficient plant, which could also be worked at a much reduced labour cost. The dryers, therefore, now usually employed, and which include the most modern apparatus, are not patented, and generally consist of revolving cylinders, 30 feet to 50 feet in length and about 4 feet in diameter. These **rotary drying drums**, as they are termed, are supported on steel tyres resting on heavy friction rolls. The drums are rotated when in use at a speed approximating two revolutions per minute, and are usually set with an inclination of about one-half inch per foot.

The raw materials after passing the crushing rolls are introduced into the upper end of the dryers, and are immediately caught by cascading channels fitted inside the drums, which lift and drop the crushed raw materials as the drum revolves, and present them to the heated gases passing through the drums in the opposite direction to that in which the materials are travelling to the lower or outlet end. The use of waste heat from the kilns is generally employed for drying purposes, or again, separate furnaces are sometimes arranged for heating the entering air at the lower end of the drum.

The raw material after thus being crushed and dried must be reduced to an extremely fine powder or "flour," and then carefully and thoroughly mixed to the proper chemical proportions before being conveyed to the kilns for burning.

If the mixture is to be burned in shaft kilns, which will be referred to hereafter, the prepared raw material must be damped to give a sufficient "bind" for making or



ROTARY DRYING DRUM—DRY PROCESS.

pressing into blocks (or bricks) to be charged into the kiln. If the rotary kiln is in use, however, the dry powder can be conveyed and fed directly into it for calcining.

The dry process of manufacturing cement is more generally employed in the United States, where hard raw materials are employed; and in the United Kingdom in cases where the interstratified limestones and shales of the lias formation supply the raw materials. In the Cambridge district, also, the chalk marls offer facilities for dry grinding, since no flints occur with these raw materials.

From the above description it will appear, therefore, that in preparing the raw materials the chalk and clay or other materials must be first reduced to the utmost fineness, either by the addition of water, which helps to break down the cohesion between the particles and leaves the material in a very finely divided state, or by the more mechanical process of grinding when dry. The physical properties of the respective materials to be dealt with will generally determine which method of reduction is to be adopted.

In the further process of **mixing** the raw materials, the ingredients are gauged in certain exact proportions, and the accuracy with which those proportions are estimated, and the mechanical efficiency with which the process of admixture is carried out, are the first essentials in the manufacture.

A dry process method, comprises firstly, machinery for crushing, and plant (such as the "tower" or drying drum here illustrated) for the preliminary drying of the raw material as it is brought from the quarry. Apparatus is then required for the reduction of the two materials to an impalpable powder, or, as we have termed it, "flour"; and mixers for the storage and mechanical mixing of the flour until passed as chemically accurate and ready for burning.

To weigh up then the specific advantages of the wet and dry processes of preparing the raw materials for cement manufacture, it should be pointed out that some materials —such as the Thames chalk—demand the wet process, so as to eliminate the flints, which cannot be entirely got rid of by other means. On the other hand, hard or soft raw materials containing no foreign matter can be ground by mills either in their wet or dry state, the economic feature

being decided by the cost of drying and grinding the raw materials (if by the dry process) and the cost of wet grinding the same materials *plus* the additional coal required



View of Raw Material Grinding Mills—Dry Process.

to burn off the excess of moisture when ultimately burning the materials in the kiln, an excess fuel up to 2 cwts. per ton of cement being necessary in the wet method over the dry method if rotary kilns are employed for burning.

If the reduction of the ingredients be completed by either of the processes above mentioned, and, chemically considered, the lines laid down be strictly adhered to, the resultant cement should be a perfect product.

The composition of the raw material mixture is ascertained by analyses and tests, undertaken by the works' chemists at frequent intervals, and the carbonate of lime (familiarly known by its chemical formula, $CaCO_3$) should be kept within, at most, one-half per cent. of the quantity found to produce the best cement. This percentage of lime carbonate varies in different works according to the raw materials obtained from the many geological formations contributing to the manufacture, but, roughly, a combination containing from 75 to 77 per cent. of $CaCO_3$ will produce a cement of good quality.

When the remaining portion of clay runs too high (or the percentage of lime carbonate falls below 75) a compound is obtained which, in the burning process, will fuse at a temperature lower than that required for the production of sound clinker, thus rendering it more or less useless. On the other hand, an excess of chalk (above, say, 77 per cent.) will allow a mixture to sustain a high temperature in the kiln without risk of fusion, but the resultant clinker would be of very doubtful quality because of its tendency to expansion when ground for cement.

Again, a variation in the amount of carbonate of lime, even to one-half per cent., is found to alter appreciably the tensile strength of a cement. A series of tests bearing on this subject were recently made with chalk marl as a raw material, and it was shown that from a perfectly mixed raw material, of 76.5 to 77 per cent. $CaCO_3$, a cement was produced the tensile strength of which at seven days was 780 lbs. per square inch, and at twenty-eight days 940 lbs. With a mixture of 76 to 76.5 per cent. lime carbonate, the strain was 610 lbs. in seven days, and 800 lbs. in twenty-eight days, and with a mixture of 75.5 to 76 per cent. $CaCO_3$, 520 lbs. in seven days and 730 in twenty-eight. All of the samples produced were perfectly sound under the strictest tests, but if the $CaCO_3$ were increased above 77.0 per cent., the cement was unsound, and similarly with a mixture of 74 per cent. $CaCO_3$, the resulting cement had little strength. This illustration is given as showing the necessity for mixing as closely as possible to definite standards of composition.

The mechanically-mixed raw material prepared in the ordinary course of manufacture may analyse as follows :—

Silica	15.72 per cent.	Clay.
Alumina and oxide of iron ...	6.10 ,,	
Undetermined	.57 ,,	
Carbonate of lime	76.50 ,,	Chalk.
Carbonate of magnesia ...	1.11 ,,	

From such a material, if properly treated in the further stages of manufacture, a good commercial cement, testing at least 700 lbs. per square inch in seven days, should be produced.

The chemist's services, therefore, are requisitioned to check the raw material mixtures both before they are quarried and after they are further dealt with, by subjecting the chalk and clay in its amalgamated state to strict analyses and tests. Of course, experience and practice in the treatment of the raw material obviate to some extent the necessity of continually analysing the mixture, but even this is systematically carried out at varying periods, according to the variation of the raw material constituents and the percentages found to produce the best quality of cement.

Estimation and Analysis of Raw Materials.

CHAPTER VI.

In the primary stage of the manufacture of Portland cement it is necessary to be chemically accurate in the estimation of the proportion of carbonate of lime to that of silica and alumina contained in the raw materials.

If the ingredients of the mixture are not prepared so as to contain the exact and necessary proportions of these compounds before the succeeding stage of manufacture is proceeded with, no effort on the part of the manufacturer and no scientific operation can bring the resultant cement up to the standard it might have attained had the proportioning of the ingredients been accurately carried out.

It is, therefore, desirable for the manufacturer to exercise an incessant check, at least upon the determination of the calcium carbonate (provided the raw material mixture is of normal constitution), and methods must be adopted of accurately testing the mixture with speed and facility, so often each day has the test to be made.

The raw material which will produce the **lime** (the main factor in the manufacture of Portland cement) is found in many places and is of many qualities. It may be in the form of slag, alkali waste, soft chalk, or a hard crystalline limestone, or one of the many grades of calcium carbonate between the two latter. As a rule, the soft chalks are very pure carbonate of lime, but they contain much water, and the hard limestones often contain in a natural state as much as 99 per cent. carbonate of lime.

The **clay** (containing the silica and alumina) varies almost as much, from soft river mud to hard shales or slate. The chalk and limestone generally contain few impurities, but the clay is apt to contain magnesia, sulphur and alkalies, the presence of which must be kept within certain strictly defined limits.

It is necessary, therefore, first to determine accurately the exact composition of the materials, as some substances are injurious to the finished cement, and others simply increase the bulk without any appreciable benefit. The smaller the quantity of these foreign substances present in the raw material the better will be the quality of the finished product.

Carbonate of **magnesia** is nearly always present in the raw material. The presence of magnesia in Portland cement is generally considered harmful if it amounts to more than three per cent.; it is, therefore, usually kept within this limit. The effect of magnesia in cement is to cause expansion, but the exact chemical action has not yet been definitely explained.

Investigations have shewn that five per cent. of magnesia mixed with good cement and made into a pat, showed no signs of expansion until the end of six months, the sample being under water all the time. Another sample experimented with was boiled for six hours before any expansion took place, and the expansion continued for thirty-four hours. Some experts consider that magnesia, though possessing marked hydraulic properties when ignited alone, yields no hydraulic products when heated with silica, alumina or clay, and probably plays no part in the formation of the cement. It is, however, incapable of replacing lime in cement mixtures, the composition of which should be calculated on the basis of the lime only without regard to the magnesia present.

Nearly all clay contains **sulphates** in small quantities, and the presence of even the smallest quantity is injurious in a theoretical sense, but in practice may not be noticeable if under two per cent. In the finished cement it may appear as sulphate of lime, commonly known as **gypsum** or plaster of Paris. This substance, in the presence of free lime, the aluminates usually found in cement, and the water used in gauging, forms with them a crystalline compound which occupies a much larger space than the aluminate and sulphate of which it is composed. For this reason any considerable quantity of sulphate will cause expansion of the whole mass of the cement, tending to produce fine cracks and materially weaken the finished work.

Should the cement contain no alumina, the dangerous

a fixed proportion in the calcium carbonate, the percentage of lime can be calculated from the volume of gas given off.

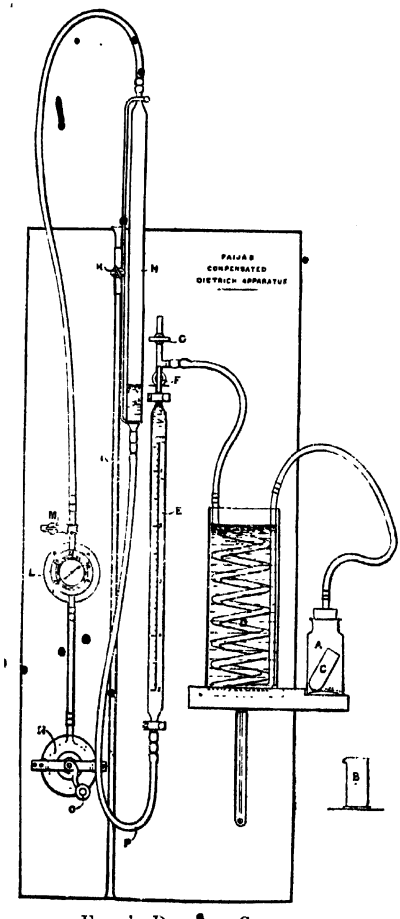
We will here give a description of the **Faija's instrument:—**

A is the generating bottle.
B is the acid measure.
C is the gutta-percha acid tube.
D is the condenser with lead coil.
E is the gas measuring tube, having at its upper end the two taps F and G.
H is the equilibrium tube, which may be moved up and down and secured in any position by fixing it on the rod I, by means of the thumb-screw K.
L is the barometer, to which is attached on its upper side, the tap M.
N is an india-rubber ball, attached by a tube to the barometer, which ball may be compressed or expanded by turning the handle O, thus adjusting the pressure in the instrument.

To set up the instrument it is first necessary to secure it firmly against a wall, taking care that the tubes E and H are perfectly vertical. Then attach the india-rubber tube P, connecting the lower ends of tubes E and H. Tube H should then be lowered until its upper neck is a little above the lowest reading on tube E, a funnel inserted in the neck of H, and the tube filled with water until the water rises up to the lowest reading in tube E. When filling with water, the two taps F and G should be open, and as air is likely to accumulate in the india-rubber tube P, this should be squeezed and pressed until the air is all expelled.

The water used should be distilled water, and in order that all air may be expelled from it, it should be boiled and allowed to cool before pouring into the instrument. Then attach the other india-rubber tubes, as shown in the drawing, put the stopper into the generating bottle A, close taps G and M, and see if the instrument is tight. This is ascertained by placing the equilibrium tube H in such a position that there is a difference of several inches in the level of the water in the two tubes E and H, and noting the reading of the level of the water in tube E; if

the instrument is tight, the water will remain at this level for an indefinite period. If, on the other hand, the water



FAIJA'S DIETRICH CALCIMETER.

in E rises or falls, some of the joints are not tight, and they must be made tight by binding them with thin brass

or copper wire. The condenser D should be filled with water.

To work the instrument.—Having ascertained that the instrument is tight, the mode of working is as follows:—

First remove the cork from the generating bottle A and take out the acid tube C, then open the taps F, G, and M. Elevate tube H to such a height that the water in tube E is exactly level with the mark immediately under tap F, and secure it there by turning the thumb-screw K. Weigh out the quantity of carbonate of which it is desired to determine the carbonic acid, and place it in the generating bottle A.

Measure out the proper quantity of acid to use in the measuring glass B, and pour it into the acid tube C. Wipe the outside of the acid tube C, so as to be sure that no acid has run down the side, and insert it, with a pair of tongs, into the generating bottle A. Re-insert the stopper in the generating bottle A, taking care that it is secure and tight. Close tap G, and take hold of the thumb-screw K with the left hand and slack it, at the same time keeping tube H approximately in its elevated position. Now take the generating bottle A in the right hand and incline it so that the acid runs out of the acid tube C on to the carbonate in the bottle A, and as the gas is generated, lower tube H, so as to keep the water in tubes E and H approximately at the same level. Continue shaking the bottle while the gas is generating, and be sure that all the acid has run out of the acid tube C. Place the generating bottle A in the water in the condenser D; this is to cool the gas, which might have been heated by the handling of the generating bottle A, down to the temperature of the water in the condenser D. Having left it there for a minute or so, remove it, and again shake it, and note if any more gas is generated. When all the gas is generated, which is indicated by the water in tube E remaining in a constant position, close the taps F and M, turn the handle O, actuating the ball N, in either one direction or the other, so that the pressure in the barometer is diminished or increased, until it indicates the normal pressure of 29.92 inches, or 760 millimetres, which

is more distinctly shown by the heavy line. Adjust tube H so that the water in it is exactly level with the water in tube E, and take the reading of the level of the water in tube E. This reading is the amount of gas developed at the standard pressure, and simply requires correction for temperature, which may be ascertained by tables already prepared.

When using the instrument the thermometer should be placed and left in the water in the condensing vessel D, as it is the temperature of this water which governs the temperature of the gas, but it is as well to try and adjust the temperature of the water to approximately the temperature of the atmosphere of the room in which the instrument is worked.

Before commencing an experiment, the reading of the barometer should be ascertained, and if above 29.92 inches or 760 millimetres, the ball N should be deflated, so that by turning the handle O, and allowing the ball to expand, the pressure in the barometer will be decreased. If, on the other hand, the barometer is below 29.92 inches or 760 millimetres, the ball N should be left fully expanded, when by turning the handle O, so as to compress the ball, the pressure in the barometer will be increased.

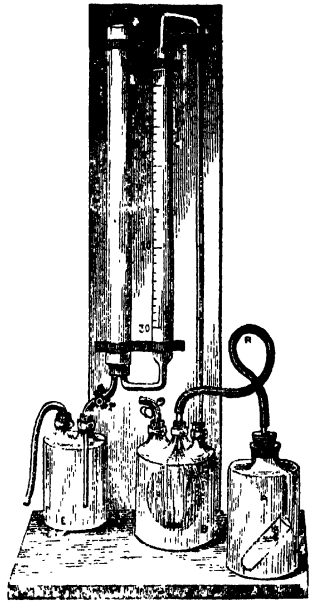
The essential portion of the Faija's apparatus for the determination of carbonic acid gas consists in the addition of a barometer to the equilibrium tube, the pressure in which, and in the gas-generating tube, may be adjusted to a standard pressure.

By this arrangement, temperature is the only factor which it is necessary to consider when the volume of the gas has been ascertained by the reading of the instrument; and for easy use with this instrument complete tables have been arranged, so that the operation of testing raw materials only occupies a few minutes.

This apparatus has been designed to obviate as far as possible the disadvantages entailed by the difference in volume of gases due to variation of temperature and barometric pressure necessitating in all instruments designed for their volumetric determination a considerable amount of calculation.

The **Scheibler's calcimeter** here illustrated consists of the following parts:—A small bottle A provided with

a perforated stopper. In the bottle is placed a tube S, of gutta-percha or glass. Another bottle B, is provided with three openings in its neck. The central opening of the bottle contains a firmly fixed glass tube, which connects



SCHEIBLER'S CALCIMETER FOR THE VOLUMETRIC ESTIMATION OF CALCIUM CARBONATE.

at the one end with A by means of the flexible rubber tube R, and at the other inside the bottle B, with a very light india-rubber bladder.

The left-hand opening is controlled by a pinch-cock on a piece of rubber tubing. The right-hand opening connects B with the measuring tube, which is an accurately graduated glass cylinder C, of 150 c.c. capacity. Another

glass cylinder D serves to regulate the pressure of the gas measured in C, and a tube P passes through a stopper into the water reservoir E, and is connected with D and controlled by meaps of a pinch-cock.

For determining, then, the amount of carbonate of lime in the raw material mixture, a small sample, say, .5 gramme, finely powdered, is placed in the bottle A. The tube or cup is filled with 10 c.c. of dilute hydrochloric acid and placed also in the bottle.

Shake the bottle A so that the acid gradually mixes with the sample, and release the cock P in order that the water which has been previously filled into the cylinders D and C may be released as the gas in C displaces the same; keep the water in D on an exact level with C. Then take the reading on C and compare the volume of gas given off from the sample with the results from testing a standard sample of pure calcium carbonate (Iceland spar).

For example, take .5 gramme pure calcium carbonate, and say the gas registers 18 c.c. on the cylinder C, and say .5 gramme of the mixed raw materials for the manufacture gives 13 c.c. of gas; the result is $\frac{100 \times 13}{18} = 72.2$ per cent. of carbonate of lime in the sample.

The apparatus for estimation is always placed in the laboratory, where direct sunlight cannot fall upon it, and is also protected from any other heat, so that the results are not affected by these conditions.

Although other methods can be adopted for estimating the carbonate of lime in raw materials, those described above are found to be sufficiently accurate for general practical purposes, and little or no technical skill is required in their manipulation.

By the **titration** test the calcium carbonate is decomposed by a measured quantity of standard nitric or hydrochloric acid, and the excess of acid determined by titration with standard alkali. This latter test is of a more technical description, and necessitates the careful preparation and standardising of solutions by qualified chemists.

The method for the estimation of carbonate of lime by titration is based on the chemical action of a given quantity of acid of known strength on a given weight of carbonate of lime; the excess of acid being found by titration with

standard alkali. The solutions required for the titration test are normal hydrochloric acid and semi-normal sodium hydrate.

To prepare a Standard Normal **Hydrochloric Acid** (HCl) **Solution**:—

Take 200 c.c. of pure concentrated acid, and well mix with 1800 c.c. distilled water.

To **standardise** the solution work as follows :—

Weigh out accurately 1.06 grammes of pure dry Sodium Carbonate (Na_2CO_3), which should be ignited and cooled in a desiccator before weighing. Transfer to a 200 c.c. flask and dissolve in about 25 c.c. distilled water. Add two drops of Methyl Orange indicator, and titrate the contents with the acid solution from a graduated burette.

It will be found that it takes 20 c.c. normal standard acid to neutralise the 1.06 grammes Na_2CO_3. Should the acid be too strong water is added, if too weak, more acid is added, but it is better to make sure of the acid being too strong as it is much quicker to dilute to Standard than to raise by addition of more acid. For rapid correction the following calculation is advised :—

Example:—Acid required to neutralise 1.06 gr. Na_2CO_3 = 19.2 c.c. As standard acid takes 20 c.c. to neutralise 1.06 gr. Na_2CO_3 each 19.2 c.c. in stock solution will require .8 c.c. water to make up to 20 c.c. The stock solution of acid is 2,000 c.c., as above mentioned.

Thus :—

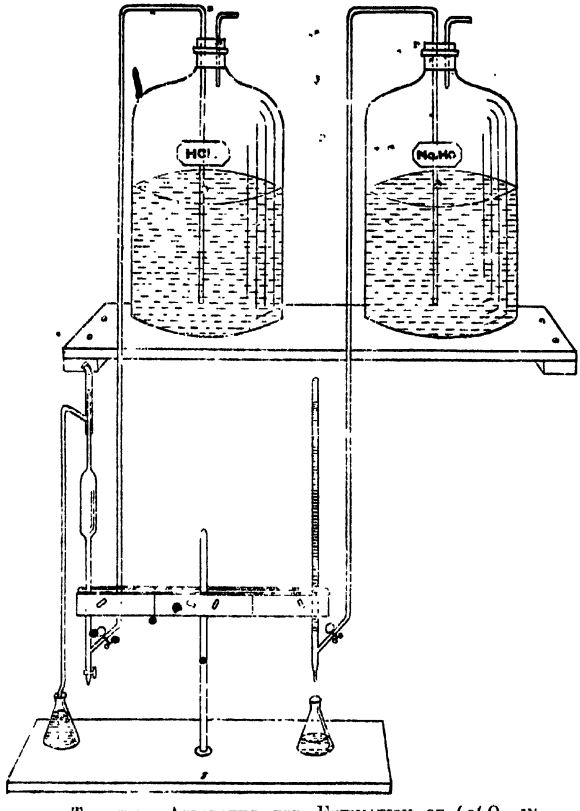
$$\frac{2000}{19.2} = 104.1$$

$\therefore$ 104.1 × .8 = 83.2 c.c. water required.

After additions have been made to the acid solution, repeated titrations with 1.06 gr. Sodium Carbonate must be carried out, showing well agreeing results at 20 c.c. before the acid is passed as correct normal.

To prepare Standard Semi-Normal **Sodium Hydrate** (NaHO) **Solution** take about 44 grammes Pure Sodium Hydrate (NaHO) sticks, and dissolve in about two litres of distilled water.

Fill a graduated burette with the Standard Acid solution, also a burette with the Sodium Hydrate solution.



Titration Apparatus for Estimation of $CaCO_3$ in Raw Material.

Run into a flask from the burette 25 c.c. of Acid; two drops of Methyl Orange are added, and then the solution of Sodium Hydrate is run into the flask from the burette

till the colour just turns yellow. As the alkali is half the strength of the Standard Acid, 25 c.c. acid should require 50 c.c. of the alkali solution to neutralise it. A strong solution of the Sodium Hydrate solution is best diluted down in the same method as used for the acid. Several titrations must be done with results agreeing at 25 c.c. acid, requiring 50 c.c. alkali before the solution is passed as correct.

In using the Standard Acid and Alkali method for **testing for** $CaCO_3$, the raw material mixture (chalk or slurry) is dried and well ground in a mortar.

Weigh out accurately 1 gramme, and transfer to a 200 c.c. flask.

From the burette run in 25 c.c. of the standard acid, wash down with some distilled water the sides of the flask, then place on a hot plate and boil for a few minutes. Remove and add to the flask a few drops of Phenol Phthalein solution (indicator); now run in the standard alkali from a graduated burette until the solution turns a purple tinge (this point, when all the acid is neutralised, is very distinct), then take the reading on the burette.

The method of calculating the $CaCO_3$ is as follows:— The number of c.c. alkali required, divided by two, gives the amount of acid neutralised by the alkali; which, subtracted from 25 c.c. gives the amount of acid used by the carbonate of lime in the slurry. This result, multiplied by five, gives the percentage of Carbonate of Lime. The above calculation is arrived at from the chemical reactions in the following equation:—

$$2HCl + CaCO_3 = CaCl_2 + H_2O + CO_2$$

An explanation of this equation is as follows:—

$$\frac{2\,HCl}{2\,(1 + 35.5)} + \frac{CaCO_3}{40 + 12 + 48} = \frac{}{73} \quad \frac{}{100} = \&c.$$

The molecular weight of Hydrochloric Acid (HCl) is 36.5 and of Carbonate of Lime ($CaCO_3$) 100. Then 100 $CaCO_3$ requires 73.0 of HCl. Normal Standard HCl contains 36.5 grammes Hydrochloric Acid in 1,000 c.c., therefore 1,000 c.c. will decompose 50 grammes of Carbonate of Lime ($CaCO_3$), therefore 20 c.c. Hydrochloric Acid will

decompose 1 gramme Carbonate of Lime ($CaCO_3$). Thus :—

Example:—1 gramme of cement raw material mixture, treated with 25 c.c. of Normal Hydrochloric Acid (HCl) and boiled, on titration takes 19.6 c.c. of Semi-Normal Sodium Hydrate (NaHO) to neutralise.

As 1 c.c. acid requires 2 c.c. alkali then 19.6 c.c. divided by 2 equals 9.8 as the amount of acid neutralised by the alkali.

Then 9.8 c.c. subtracted from 25 c.c. (the amount of acid taken) leaves 15.2 c.c. which have been used up by the Carbonate of Lime.

Now :—

	20 c.c. acid	=	1 of Carbonate of Lime	
	15.2 c.c. ,,	=	.76 ,,	,,
	In 1 gramme of mixture there is		.76 ,,	,,
∴	,, 100 grammes ,, ,,		76.0 ,,	,,
		=	76.0% $CaCO_3$	

or the equation is as follows :—

$$\text{Acid} - \frac{\text{c.c. of alkali}}{2} \times 5 = \% \text{ of } CaCO_3$$

In a well-appointed establishment, if the raw materials are found to vary in analysis, the testing takes place as the materials are quarried, and the proportions are again corrected before the mixture is passed for the succeeding process of calcination—a stage, again, entirely dependent upon the accurate performance of these chemical tests, and upon a due appreciation of the meaning and value of their results.

Analysis of Raw Materials.

When a mixture of the raw material has been prepared for the manufacture of Portland cement the method of analysis is as follows :—

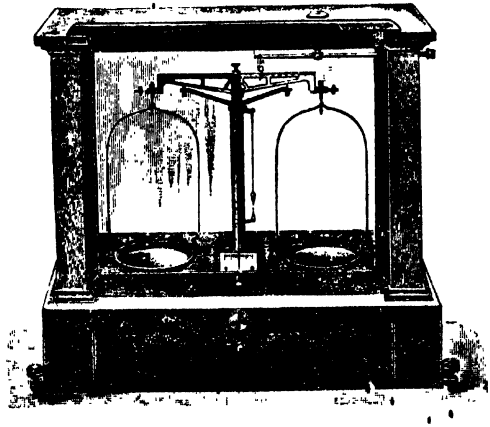
Loss on Ignition.—Heat 1 gramme of the mixture in a platinum crucible, first gently over a bunsen flame, then strongly over a blast lamp. Cool in desiccator and weigh. The difference in weight is the loss on ignition

in organic matter, carbonic acid gas, and any combined water.

Insoluble in Hydrochloric Acid (10 per cent.).—Now break up the ignited material in the crucible with a smooth glass rod. Transfer to a beaker and digest with a 10 per cent. solution of Hydrochloric Acid.

Filter off insoluble material, and wash well with hot water; ignite and weigh.

Silica (SiO_2) **and Chief Bases**:—The insoluble residue from the above is then mixed thoroughly with



Chemical Balance.

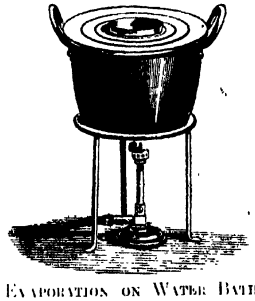
three to four times the quantity of double carbonates (Sodium and Potassium Carbonates—Fusion Mixture). Heat the crucible gently at first over a bunsen flame, gradually increasing the flame, then to a quiet fusion over a blast lamp.

This fusion decomposes the insoluble silicates.

Allow the crucible to cool to dull redness, then place it upright in cold water to induce rapid cooling and cause the mass to leave the sides of the crucible. Place the crucible and contents in a beaker with 50 to 100 c.c. of warm water, cover with a clock glass, and digest for a few minutes.

Now slowly add strong Hydrochloric Acid (HCl) drop by drop till the solution is acid, and digest until all the contents are dissolved. Pick out the crucible, clean with a rubber-tipped rod and wash well. Now pour the contents of the beaker into an evaporating dish, and mix together with the filtrate from the insoluble in Hydrochloric acid (above). Then place on a hot plate and evaporate to dryness, but care must be taken against loss from spurting. If desired or necessary the analysis of the soluble and insoluble in hydrochloric acid may be carried out separately.

When dry, bake the contents for ten to fifteen minutes at a good heat to make the silica (SiO_2) insoluble. Cool, add 50 c.c. Hydrochloric Acid (1 to 1 of water) and digest

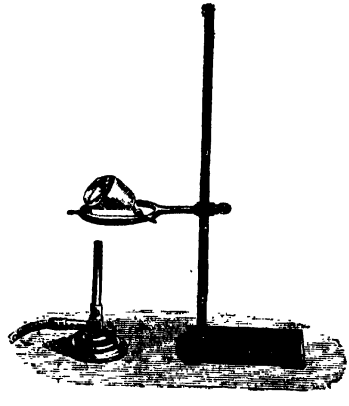


Evaporation on Water Bath.

for a short time, then add about 50 c.c. warm water; boil and filter off the silica. As some silica generally remains soluble where there is a large body of salts from an alkaline fusion, it is best to again evaporate the filtrate to dryness and dissolve up as before, filtering the solution through the same filter paper. Wash the precipitate well with warm water to free it from Alkali Chlorides. The moist precipitate and filter paper are placed in a platinum crucible, ignited gently to burn off the carbon, then strongly over a blast lamp. Cool in a desiccator and weigh as Silica (SiO_2).

Oxide of Iron and Alumina (Fe_2O_3, Al_2O_3).—To the filtrate from the Silica add a few drops Nitric Acid (HNO_3) and boil; now add slowly a slight excess of

ammonia and boil till the liquid has only a faint smell of ammonia. Filter off the precipitate and wash once with hot water, wash the precipitate back into the beaker and dissolve in Hydrochloric Acid (1 to 1 of water) and re-precipitate with ammonia, boil as before and filter off the precipitate, allowing the filtrate to run into the one from the first precipitation; then wash well with warm water. The moist precipitate and filter paper are then transferred to a Platinum Crucible, ignited gently first, then with



IGNITION OF PRECIPITATE IN PLATINUM CRUCIBLE.

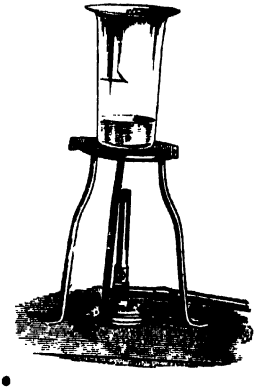
full flame of blast lamp. Cool and weigh as Ferric Oxide (Fe_2O_3) and Alumina (Al_2O_3).

Separation of Iron and Alumina (A) AND (B).—A. The precipitate after weighing is fused up with a little sodium carbonate (Na_2CO_3), the mass dissolved in a little hydrochloric acid, a strong solution of caustic potash (KHO) added and the solution boiled for five minutes. Dilute the solution slightly and filter off the iron precipitate and wash well. (The filtrate (X) is laid aside and used for the determination of the alumina.)

The iron precipitate is dissolved in hydrochloric acid (HCl) and re-precipitated with ammonia, boiled, filtered,

well washed, ignited and weighed as ferric oxide (Fe_2O_3). To the filtrate (X) add hydrochloric acid, till the solution just shows an acid reaction with litmus paper. Add a slight excess of ammonia, and boil until the liquid has only a faint smell of ammonia. Filter off the precipitate, wash well with hot water, ignite gently at first, then strongly in blast lamp; cool and weigh as alumina (Al_2O_3).

B. Instead of burning and weighing the combined precipitate, the precipitate is washed back into a beaker and boiled direct with caustic potash (KHO) for the separation of the alumina, the method being exactly as for A.



Boiling Solution for Precipitation.

Lime (CaO).—To the combined filtrates from the two precipitations of the iron and alumina, add a few drops of ammonia and bring to boiling, then add about 25 c.c. of a saturated solution of ammonium oxalate, stir well, and boil for one minute. Allow the precipitate to settle, then decant as much of the clear solution as possible through a filter paper, keeping the precipitate in the beaker. Now dissolve the precipitate in dilute hydrochloric acid and boil, re-precipitate with ammonia and a little ammonium oxalate. Allow the precipitate to settle and filter through the same filter paper, allowing the filtrate to mix with the first one; wash the precipitate with a little warm water, replace the

beaker under the filter funnel for the remainder of wash water which is ignored. (The filtrate from first and second precipitation together with the first wash water, is kept for the magnesia determination). The precipitate is all washed into the filter paper and allowed to drain, the moist precipitate and filter paper is transferred to a platinum crucible, ignited gently till all the carbon is burned off, then strongly ignited in the blast lamp till the weight is constant. The calcium oxalate is reduced on heating to lime (CaO) and is weighed as such.



FILTERING OFF A PRECIPITATE.

Magnesia (MgO).—The filtrates from the lime are cooled down, and an excess of ammonium phosphate solution is added and stirred briskly. Then about 10 c.c. strong ammonia is added, the solution is again stirred briskly and allowed to stand overnight.

The precipitate is then filtered off, and washed with cold dilute ammonia water. The moist precipitate is then ignited, first gently, afterwards in the blast lamp, and weighed as magnesium pyro-phosphate.

To calculate the magnesia, multiply weight found by .3624 which equals MgO.

Sulphuric Anhydride (SO_3).—Two grammes of the raw material are boiled with hydrochloric acid (1 to 2 of water) and filtered. The filtrate is raised to boiling and a 10 per cent. solution of barium chloride is added, and the whole boiled for a few minutes. The precipitate of barium sulphate ($BaSO_4$) is allowed to settle for two hours, filtered, well washed with warm water, ignited and weighed. Calculate as follows:—Barium sulphate ($BaSO_4$) found, divided by two, multiplied by .3433 gives sulphuric anhydride (SO_3).

The Alkalies sodium and potassium are not ordinarily determined.

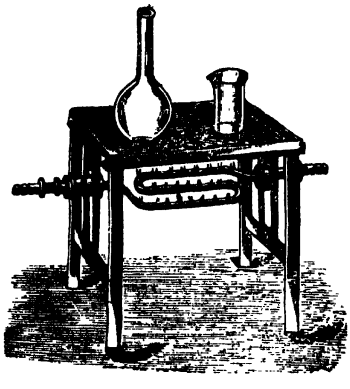
An analysis of chalk is conducted as follows:—

Silica and Insoluble.—Weigh out one gramme of finely ground dry chalk into an evaporating dish, add about 30 c.c. water and cover with a clock glass, then slowly add about 10 c.c. concentrated hydrochloric acid (HCl). When all effervescence has ceased, wash the clock glass into the dish, place the dish on a hot plate and evaporate the contents to dryness. Bake for 15-20 minutes to drive off completely the hydrochloric acid and make the silica insoluble. Cool the dish, add about 25 c.c. hydrochloric acid (1 to 1 of water), and digest on hot plate or sand bath for a few minutes, add about 50 c.c. warm water and boil, filter, wash the precipitate in filter paper well with warm water, ignite strongly on blast lamp, and weigh as silica (SiO_2) and insoluble. If necessary to estimate the silica, fuse up the silica and insoluble with 8-10 times the quantity of double carbonates and proceed with analysis as in cement mixture.

Ferric Oxide (Fe_2O_3) **and Alumina** (Al_2O_3).—To the filtrate add ammonia in excess, boil till solution only smells faintly of ammonia, allow precipitate to settle and filter off, washing once with hot water, dissolve the precipitate in dilute hydrochloric acid and re-precipitate with ammonia, boiling as before. Filter, wash well; allowing the filtrates from both precipitations to mix, ignite strongly and weigh as ferric oxide and alumina.

If necessary to separate the iron and alumina, follow method given in analysis of cement mixture.

Lime (CaO).—To the combined filtrates add a few drops of ammonia and raise to boiling, then add about 25-30 c.c. of saturated solution of ammonium oxalate, stir well, and boil for one minute. Allow the precipitate to settle, then decant the clear liquid through a filter paper, keeping the precipitate in the beaker. Now wash the precipitate in the beaker with warm water and filter off.



Hot Plate or Sand Bath for Evaporation.

washing with warm water into the filter paper. Allow to drain. Ignite gently first, then in the blast lamp until the weight is constant, weighing as lime (CaO).

Magnesia (MgO).—To the filtrate from the lime add excess of saturated solution of ammonium phosphate, stir well, then add about 10 c.c. strong ammonia, stir again, and allow to stand over night. Filter off the precipitate, wash well with dilute ammonia water, ignite strongly and weigh as magnesium pyrophosphate ($Mg_2P_2O_7$). Calculate as previously.

The **alkalies** are not usually determined.

An analysis of clay is conducted as follows :— Grind up the sample of clay very finely and dry in an oven at 100°-110°C. Weigh out one gramme into a platinum crucible and mix well with 8 to 10 times the quantity of double carbonates (sodium and potassium, Na_2CO_3, K_2CO_3, fusion mixture).

Heat gently at first over a Bunsen flame, then ignite over blast lamp to quiet fusion. Allow the crucible to cool below redness, then place upright in cold water to induce rapid cooling and cause the melt to leave the crucible. Now place the crucible in a beaker with about 40 c.c. hot water and digest for a few minutes. Cover the beaker with a watch glass and make the solution acid by adding slowly strong hydrochloric acid, digest till all the melt is dissolved, pick out the crucible and thoroughly wash.

Pour the contents of the beaker into an evaporating dish and evaporate to dryness and bake for 20 minutes. Cool the dish and add about 30 c.c. hydrochloric acid (1-2 of water) and digest on the hot plate for a few minutes. Add about 50 c.c. warm water, boil and filter off the **silica.** Transfer the filtrate to the evaporating dish and again evaporate to dryness, bake as before, then dissolve up in the same manner, and filter through the same filter paper.

Owing to the large body of salts, it is extremely difficult to get the silica baked, so two evaporations are necessary to get all the silica out of the solution. Well wash the precipitate, ignite strongly, and weigh as silica.

Ferric Oxide and Alumina.—Add excess of ammonia to the filtrate from silica to precipitate the iron and alumina. Boil off the excess of ammonia, filter and wash once with warm water, dissolve the precipitate in hydrochloric acid and re-precipitate with ammonia in same manner.

Boil to free the solution from excess of ammonia, filter, wash well with hot water, ignite and weigh as *ferric oxide and alumina.*

If necessary to separate the ferric oxide and alumina, follow the method given under the raw material mixture analysis.

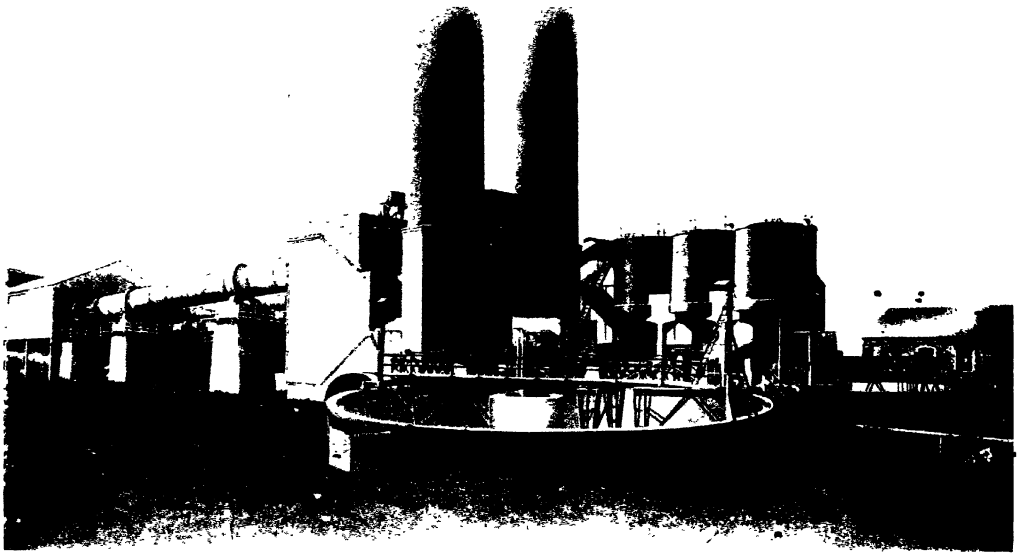
Lime.—To the mixed filtrates from the two precipitations of iron and alumina, add a few drops of ammonia

and boil, then add about 25 c.c. saturated solution of ammonium oxalate, stir and boil for one minute. Allow the precipitate to settle, and filter off the clear solution, keeping the precipitate in the beaker; now dissolve the precipitate in hydrochloric acid and re-precipitate as above with ammonia and a little ammonium oxalate, boil, allow to settle, and filter through same filter paper. Wash well with warm water, allow to drain, ignite strongly and weigh as (CaO) lime. The precipitate is ignited in the blast lamp until the weight is constant.

Magnesia.—To the mixed filtrates from the lime precipitations add excess of ammonium phosphate and stir well, then add about 10 c.c. strong ammonia and again stir, allow to stand over night. Filter the precipitate, wash well with dilute ammonia water, ignite strongly and weigh as magnesium pyrophosphate ($Mg_2P_2O_7$). Calculate as previously.

The **alkalies** are not usually determined.

For the estimation of **sulphuric anhydride,** proceed as in the analysis of the mixed raw materials.



Rotary Kiln Plant with Slurry Storage

Burning or Calcining.

CHAPTER VII.

THE stage in the manufacture of Portland cement which follows the scientific and mechanical preparation of the raw materials is that of calcining or burning the raw product at a high temperature to a point of slight vitrifaction, resulting in what is usually called a cement "clinker."

As will be seen from the foregoing chapters, Portland cement consists of a chemical combination of chalk (carbonate of lime) and clay (silica, alumina, &c.) which are fused together under the action of great heat, resulting in a clinker which when ground to an impalpable powder is ready for the market as Portland cement.

In the raw materials it will be observed that the carbonate of lime, silica and alumina are in a chemically uncombined state; in the conversion of the prepared raw materials into cement clinker by burning, the silica and alumina of the clay immediately enter into combination with the lime, forming calcium silicates and aluminates.

These compounds are the important constituents of Portland cement, and give to it, when combined in their proper proportions, its hydraulic properties. None of the cements of commerce, however, is made up wholly of these three ingredients, for the raw materials from which cement is made are never quite pure. It has been contended, for instance, that iron oxide behaved, in burning a mixture, the same as alumina, and that a good cement could be made in which all the alumina was replaced by iron oxide.

The calcining process is a purely chemical one, and a great responsibility is involved in the proper burning of the raw materials; for just as the primary blending of the chalk and clay can make or mar the quality of the cement,

so may the burning of the material destroy all that is valuable in the finished product. If the temperature is too high in the kiln the resultant material is impaired as a cement; on the other hand, if the heat be not sufficient, the necessary chemical changes do not take place, and a similarly unsatisfactory result is obtained.

The proper degree of burning is indicated by the formation of a dense greenish-black clinker. Light-burned clinker is brown in colour, and comparatively soft, while over-burned clinker is fused and slag-like. Long-continued burning, excess of clay, or imperfect raw mixing causes the clinker to "dust" or fall to powder on cooling, and this powder shows little or no hydraulic properties. This defect is much more common in vertical or intermittent kilns than with Rotary kilns, as the quick burning and rapid cooling by the latter process is most beneficial to the clinker.

Little information is published concerning the critical **temperature** in the process of calcination, chiefly owing to the difficulties of obtaining accurate records in practice. Laboratory experiments have, however, been made from time to time, and these tests show that the temperature obtained in the kiln for producing normal Portland cement (75 to 76 per cent. $CaCO_3$) is close to 1,400° C., or 2,552° F. A reduction of 3 per cent. in carbonate of lime has reduced the calcining temperature some 50° C., or to 2,462° F., but these temperatures are only approximate.

It is generally considered, however, that for the heat usually required to bring the raw materials to the point of incipient fusion a temperature of some 2,500° F. must be maintained in the kiln.

This temperature is variable, according to the analyses of the raw materials and particularly the percentage of carbonate of lime contained in them; and, again, it has been further shown by experts that with cement of normal composition the finer the grinding of the raw mixture, and the more thorough the burning, the more complete the diffusion, and the less tendency there will be towards a tension in the solid solution. As an illustration of this, it will be known that clinker prepared from raw materials of ordinary fineness will sometimes set rapidly when first ground into cement. If the same raw mixture is ground to a much finer powder and burned

in the laboratory, a clinker is obtained which yields a cement that does not set in less than one hour after being made. At the same time, such very fine raw materials can be burned at a temperature more than 200° F. below that required for the coarser industrial mixture, and if burned at the usual high temperature very finely ground raw materials fuse quite readily. This leads to the conclusion that a proper balance between fineness of grinding and fuel expenditure must be arrived at in order to effect the greatest economy.

In the district where fuel is cheap, a coarser raw mixture may be burned than would be the case where fuel is dear, and in the latter case it would be cheaper to go to a greater expense for finer grinding. On this basis we arrive at the theoretical conclusion that if a very finely ground mixture were submitted to a very low temperature, say 500° F. below that usually employed, for a sufficiently long time, the result would be as satisfactory a clinker as that now produced. It is probable, for instance, that with the dome kiln hereinafter explained the greater length of time of burning which is necessary is due to the fact that diffusion goes on much more slowly than in the rotary kiln, and at a lower temperature. It also explains why that portion of the clinker which has not attained so high a temperature as that in the hottest part of the kiln sometimes dusts. Diffusion has not been complete in this instance and the solid solutions are not in equilibrium.

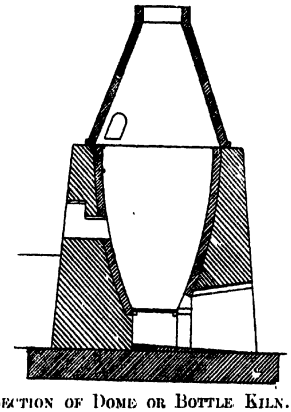
It follows, therefore (1) that the higher the percentage of lime the higher the temperature which is necessary to complete diffusion in combination with silica, and the greater the necessity for a large surface area of contact; and (2) the finer the grinding of the raw mixture the higher the percentage of lime which can be carried.

It is thus apparent that a proper chemical composition in the raw materials alone is no guarantee whatever that the cement will be a satisfactory one, as in such a case the materials of which it is composed may not have attained thorough diffusion, and, consequently, not be in equilibrium.

It is very necessary, therefore, that keen attention should be given to the proper calcination of the raw materials, a process requiring considerable experience and care.

Unfortunately, the burning of cement up to the present has not been a scientific process, and too much attention and study cannot now be given to this hitherto imperfect stage in the manufacture. It is true that, with the advent of the Rotary Kiln many facilities have been afforded for investigation in this direction, and there is little doubt that much scientific knowledge and practical improvement will result from the growing uses of this new departure in burning operations.

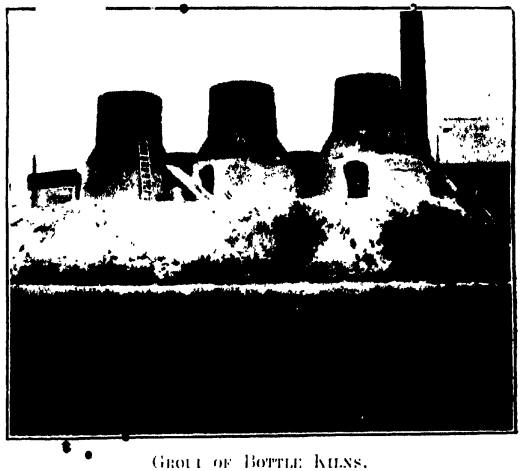
The **Kilns** for burning the raw materials are always constructed for either an "intermittent" or "continuous" process, among the former being the original Dome or "Bottle" Kilns, now obsolete, and the chamber type of Kilns; while the latter comprise the Dietzsch, Hoffman, Aalborg, Stein, and Schneider or Shaft Kilns, and, lastly, the more modern kiln now generally adopted—the Rotary Kiln.



Section of Dome or Bottle Kiln.

Intermittent kilns are of such a design that the burning necessitates a distinct loading and drawing operation, giving intermittent working and demanding the shutting down of the kiln during the operation of drawing the clinker. The loading, burning, cooling and drawing of the

intermittent kiln occupies some five to six days. In the **continuous** kilns the burning of the raw material is continuous, the " loading " of the kiln and the " drawing " of the clinker taking place at the same time, while the burning proceeds without interruption, thus making a continuous process. Continuous kilns are generally more costly to construct and require more skilled labour for their operation. These kilns are, however, economical in fuel, and where they are used the calcining process is much more regular and the cement generally of better quality.



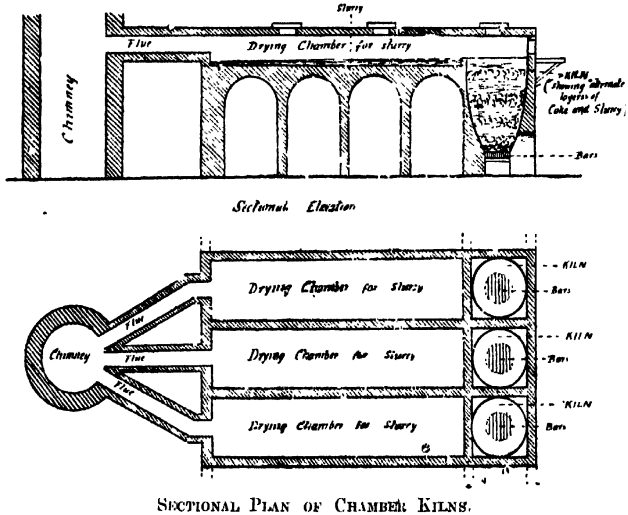
Group of Bottle Kilns.

The intermittent **Bottle** kiln was built in brickwork, vertically oval shaped, firebrick lined, having loading " eyes " near the top of the kiln. It was originally the only type employed in the industry, but is not now in use. Coke was used as fuel, and the consumption amounted to about 45 per cent. of the weight of the clinker produced. The method of working the kiln is similar to that of the Chamber types, which are still used in this country to a small extent.

The first step in loading is the careful arrangement of the coke upon the faggots which are placed on the bars of

the kiln and are used to start the fire. The kiln is then loaded up with alternate layers of raw material and coke, until filled. As the loading of the kiln proceeds, the attendant burners arrange that the layers of fuel and raw material shall gradually decrease somewhat in thickness towards the top. By this means the requisite heat is distributed more regularly over the kiln when it is fired.

In the conversion of the mixture of carbonate of lime and clay into cement clinker the following **changes** take



SECTIONAL PLAN OF CHAMBER KILNS.

place. When the coke becomes ignited by the firing of the faggots moisture from the raw material is expelled. With the subsequent red heat the carbonic acid gas is given off, and the final stage in the burning is approached when the temperature is increased to almost a white heat of 2,000° to 2,500° Fahr., causing the material to change into a state of slight vitrifaction. Layer after layer of the blocks of raw material are thus calcined into what is then termed cement "clinker." With the "intermittent" kilns above mentioned, this process goes on until the whole of

the contents of the kiln are burned through and the clinker cooled down. When cool the unloading of the kiln takes place.

The Dome or Bottle kiln, here illustrated, is of the simplest type of construction. The chamber is usually egg shaped, and as explained, the lumps of raw material and coke are passed into the kiln in alternate layers, the estimation of the proper amount of the latter requiring much experience since the draught in the kiln varies with the weather, and thus this method of burning is more or less at the mercy of the winds. Hence the amount of over-burned and under-burned clinker is likely to be excessive and the output small. The cost of labour and fuel in working these kilns is high, and therefore the kiln has become obsolete.

The **Chamber** kilns when introduced were undoubtedly a great advance on the previous system of burning for wet process manufacture, and these kilns are still used to a small extent in the wet process in this country with coke as fuel; this type of kiln is also here illustrated. The main economy in the case of the Chamber kiln is effected by the hot gases passing from the kiln over the wet slurry which has been previously pumped from the mixing plant into a chamber which is practically the flue of the kiln, the kiln proper being at one end of this chamber and the chimney stack at the other.

The Chamber kilns are intermittent and have a comparatively small output, and require considerable labour and heavy quantities of coke.

There is also some skill required in the management of the chamber kiln. If the "slip" when put into the kiln is not reasonably dry, the moisture which is driven off frequently condenses on the surface of the slurry in the chamber, adding to that already in the slurry. This water has, therefore, to be got rid of before the drying of the slurry commences. Then there is the additional danger of the slip falling away to dust in the kiln and more or less blocking the draught. Again, if the proportion of water in the slurry is not kept down as low as possible, the heat in the waste gases from the kiln is insufficient for drying, and extra fuel has to be added to the kiln.

Very often is it the experience on cement works that too little care is given to the question of the proportion of water in the slurry, and as this is a material factor in the process of manufacture attention should be given to this point.

It is fairly certain, however, although not usually realised, that it is advantageous with these kilns to carry away the resulting gases and steam, without passing them over the slurry until the kiln "drops," as it is usually called, or, in other words, until the fire breaks its way through the material in the kiln and the contents consolidate. Only then when the hot gases are fairly dry should they be allowed to travel over the slurry, and with enough draught merely to prevent the fire from being smothered. By this means, the gases are kept back, so to speak, and held down to the slurry, which then dries very rapidly. If this is done, two feet of slurry in the drying chamber can be dried in twenty to thirty hours.

Given ordinary attention the resultant clinker from the Chamber kiln is generally of first-class quality, and a thoroughly good cement is manufactured by this process of burning, providing only good quality clinker is chosen for grinding.

The **Batchelor** type of chamber kiln is constructed on somewhat similar lines to the Chamber kiln, with this difference, that instead of the single chamber, as described in the paragraph above referring to the Chamber kiln, a series of slurry-drying chambers are arranged.

At the back of the burning portion of the kilns two or three arched chambers are arranged one above the other. The hot gases from the kilns circulate through the chambers and are finally discharged into a chimney at the rear end of the kiln, one powerful shaft serving for a block or group of kilns. This kiln may be lighted as soon as charged and the process of covering the floors with slurry may proceed during the early stages of burning.

With this kiln, the arrangement described for the working of the Chamber kiln is, however, not so easily adapted, as the gases have to be drawn down to the second level. In some cases the chamber is on one level, but divided into two compartments by a longitudinal wall. The two chambers are so arranged with dampers that either or both can be connected to the kiln and chimney. The Batchelor

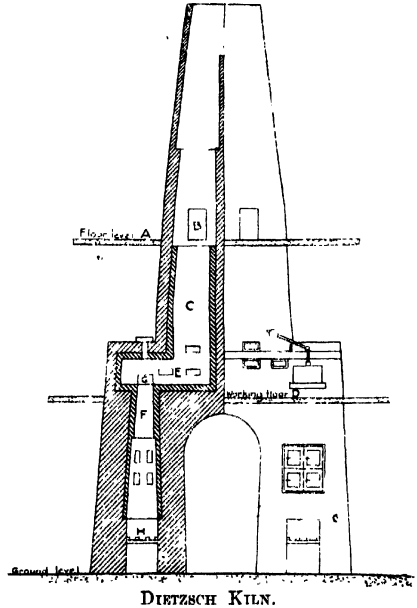
kiln is used in the wet process of manufacture in this country, but again it is costly in labour and fuel (coke), although giving good results.

With the **continuous** kilns now used in Great Britain —most of which are mentioned heretofore—the heat is kept up by the regular feeding of the coal, or coke, used for fuel together with the raw material. The principal difference between what may be described as the old intermittent method of burning, and the continuous kilns here mentioned is, that, instead of burning the kiln right out, cooling, and drawing it empty each time, the burning of the kiln rarely ceases (except for repairs), thus showing considerable economy, since the heat is constant, and the cold air in travelling up through the burnt clinker, which it cools at the bottom of the kiln, is at the same time itself heated on its way to the firing zone. After leaving the fire zone in the form of heated gases, the outgoing air parts with the greater portion of its heat in drying the raw material that is travelling downwards (towards the fire zone), and through which the hot air passes, now comparatively cool, on its way to the chimney.

The economy of a continuous kiln is proved by the low temperature of the gases when they reach the chimney, the reduction being so great that although the temperature must have been some 2,500° Fahr. at the fire zone of the kiln, the heat of the gases leaving it is only sufficient to evaporate the moisture. There is no doubt that a considerable saving of fuel (some 50 per cent.) is effected by the adoption of certain "continuous" types of kilns, for the constant cooling and heating of the dome kiln, the chamber, and other kilns previously mentioned (the interval occurring between the unloading and loading) is so much heat, and consequently so much fuel, wasted.

The **Dietzsch** kiln, now rarely employed in England, is one of the examples of the second type, or continuous kiln. The dried slurry or raw material shaped into blocks in the form of bricks is introduced at the base of the stack, into what may be called the heating chamber. Below this there is a right angle flue with a short horizontal section, from which the hot slurry is raked, so as to fall into the burning chamber which is fed with coal by hand

labour. The clinker in the lower part of the kiln is cooled by the air entering through the gates below, while the dried slurry in the upper chamber is heated by the gases escaping from the burning zone. At intervals a portion of the clinker, partially cooled, is removed at the bottom of the kiln. This causes a general settlement in the kiln and leaves a space at the top of the burning chamber, into



DIETZSCH KILN.

which the dried slurry from above is raked, and more fuel added, and so the process of burning continues. This kiln takes small or " pea nuts " size coal for fuel and is more economical than the intermittent types, using only some 25 per cent. of coal for the weight of clinker produced.

The working of the Dietzsch Kiln, illustrated on this page, may be described as follows :—

The raw material blocks are loaded on the floor level A, into the opening B, and the chimney C is thus always full.

On the working floor D, the blocks are drawn by the kiln burners over from the stage E by long shovels, and dropped into the fire zone F, and after each layer of raw material is placed in position it is covered with a layer of good quality nutty coal, and the loading door G is closed. The burned material then passes down to the bottom of the kiln, and the clinker is drawn at H. The operations of loading and drawing the kiln proceed simultaneously.

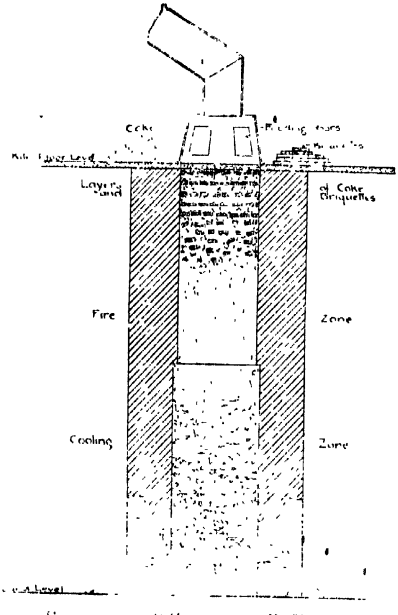
The **Hoffman** Kiln has not met with much success in this country owing to the fact that the wet process of manufacture is more generally adopted, and the kiln is essentially designed for the dry method. In this kiln small dust coal only is used, but with greater economy in quantity consumed (some 25 per cent.) as compared with the fuel consumption of the intermittent kilns previously mentioned.

The Hoffman ring kiln consists of a series of compartments built round a large central stack. The chambers communicate by means of flues in such a way that the smoke and hot gases from one may be passed through other chambers before reaching the chimney. The kiln may be either "up draught" or "down draught," according to the direction in which the heat is drawn through the chamber. The compartments are charged from the sides, and when the moisture has been driven off from the material in the chamber first fired, the gases from this chamber are passed through the adjacent chambers, which have in the meantime been filled with raw material. Although this kiln is economical in fuel if run continuously, much labour, entailing heavy costs, is required to charge and empty it.

The **Aalborg** Kiln is a shaft kiln more commonly employed on the Continent, and little, if at all, used in England. It consists of an upper heating chamber and a central combustion chamber of reduced diameter. The constructive details have been carefully arranged and the chimney is carried on the outer walls of the kiln, the inner walls allowing for the contraction and expansion in working the kiln, which thus has little, if any, effect upon the life of the kiln body.

The firing holes are placed at intervals round the kiln.

and these slant downwards so as to deliver the coal below the clinkering zone. In this kiln cement is successfully burnt, so it is stated, with an expenditure of coal in the proportion of about 20 per cent. of the clinker produced, whilst the labour involved is also small.



SECTION OF "SCHNEIDER" KILN.

The **Schneider** continuous kilns here shown—generally termed "shaft" kilns—are worked on somewhat similar lines to the intermittent kilns, but the layers of raw material and fuel are fed into the top of the kiln while at the same time the clinker can be drawn from the bottom. The fire zone occupies a position above the middle third of the cylindrical building, the layers of raw material and coke being contained in the upper portion, and the clinker in the lower.

This **shaft kiln** consists merely of a vertical brickwork cylinder some 16 feet in diameter (outside) and 40 feet in height from the ground floor to the loading floor. It is lined with firebrick for the top 20 feet, the lower half being lined with blue bricks to withstand friction from the clinker.

The inside diameter is some 9 feet, and a number of kilns are usually connected to one shaft or chimney from which the draught is obtained.

In some cases, however, each kiln emits its gases through a separate chimney some 15 or 20 feet in height.

The kiln, before being first lighted up, is loaded with bricks or stones to about half-way up the kiln, and then preparations for the fire are made by loading faggots on the stones. The loading of the coke and raw material then proceeds until the kiln is full and the fire is started through spaces purposely left in the arrangements for loading.

When once the kiln is lighted and the raw material burns through, the drawing of the " packing " commences and, simultaneously, further loading of coke and raw material layers proceeds, and the process of burning is continuous.

After about three days the clinker appears at the bottom of the kiln, and the drawing and loading continue about every four hours night and day.

This kiln has attracted some attention in this country, and is installed at several different factories, with satisfactory results. The upper heating section of the kiln is fairly capacious, but the hot zone is relatively small, and should not extend over a vertical height of more than 5 or 6 feet. Great care is taken to protect the firebricks, and this can be done by the use of a specially-contrived filling ring, which enables the burner to surround the firebrick lining with raw material, and which provides a protective belt of finely broken slurry or raw flour, about 2 inches in thickness. In practice, however, the ring is brought little into use, as the lining packing can proceed without this contrivance. The kiln is both easy to work and very economical in fuel, and large numbers of this type of shaft kiln have been erected on the Continent.

The yield of clinker may vary from 10 to 12 tons per day with natural draught, and the coke required

will run from 40 to 60 cwts. for this quantity. The kiln is not an expensive one to build in brickwork, and the cost of repairs is relatively trifling, provided care is taken with the linings of the kilns in working. The shaft kiln is in use in this country for burning the raw material bricks made by the dry process, which may be put into the kilns direct from the brick machines, and in some works which use the wet process such kilns are employed for burning surplus dried slurry from chamber or other kilns.

More recently on the Continent a new system of burning with the shaft kiln has been adopted, by which coal is used as fuel, instead of coke, and it is further claimed that a considerable saving of labour is effected, as the skilled workmen required in the loading of the original kiln can be dispensed with. By the latter process, the raw material is fed into the cylindrical kiln by mechanical means, and the alternate layers of coal and raw material are loaded and arranged by an apparatus specially designed for lowering them on to the working level of the kiln, and containing partitions into which the coal is fed. By this means blocking is prevented, and an efficient draught is thus given to the kiln. The mechanical contrivances used for loading the kiln are accompanied by devices for mechanically withdrawing the clinker, but this arrangement has not yet been adopted in British works.

Another method for working the shaft kiln is that of mixing together the raw material and the coal or coke-duff used as fuel and mechanically loading the mixture in blocks on to the existing fire. The composition of the coal in this case must be of good quality in order to burn quickly and cleanly in the clinkering zone.

The removal from the kiln of the clinker, which appears partly in small blocks and partly in pieces of the size of a walnut, is effected by small trucks arranged in front of the kiln, or by automatic conveyors. The grinding of the resultant clinker is done by the ordinary plant with comparative ease, as the porous state of the product makes it easier to grind than is the case with almost any other kiln. The saving of power necessary for grinding amounts to some 20 per cent.

The faults of the original Schneider kiln were the high labour cost (though no higher than with the other systems

mentioned) and the dependence upon coke as fuel. These drawbacks are done away with in the newer type of shaft kiln, and the advantages of the system, such as cost of plant, cheaper fuel, absence of excess labour, increased output of kilns and grinding mills, and more "flour" in the finished product, as well as the possibility of working the kiln by hand in case of machinery stoppages, all point to a prolonged life of this new kiln or, more strictly speaking, new process of burning with the shaft kiln.

In regard to this arrangement for working the shaft kiln, it might be stated generally that in laying down the plant of new works, or in changing or adopting old plants for a new process, the consideration of first importance is, of course, the type of kiln to be selected, since upon the perfect reliability of their working and fuel cost depends directly the economical success of a factory.

It is natural that every manufacturer should be on the look-out for improvements upon the many systems of kilns at present in vogue, as it should be the desire not only to simplify the working of existing kilns and to increase the output, but also to effect greater economy in fuel than is the practice to-day.

The old shaft kiln offered great advantages in these respects, not only in the smaller original cost of plant but also by a certain amount of control over the burning process, but it had these disadvantages, that it necessitated the use of a dear fuel (coke) and requires for the working of it a comparatively large and well-drilled working staff compared with the more generally employed Rotary Kiln.

Yet another system of working the shaft kiln has been evolved, and this is based upon the working of the old Schneider kilns, as described, but which it improves upon in such a way that it retains the former superiority of the shaft kiln and removes many of its drawbacks.

This is called the **forced draught shaft kiln** and which lends itself mainly to the dry process of manufacture.

The laying down of a forced draught shaft system of kilns is considerably cheaper than that of a rotary kiln, and every old shaft kiln could be, without much expense, changed into the new type of kiln, and the working of it is so simple that it may be done by untrained labour. The ground raw material requires pressing into blocks for this

kiln, but the press (often a wire cut brick machine) is arranged so simply that it does not require nearly the time, labour or power that is required for the grinding of the coal for use with the rotary kiln.

There are several designs of very simple machines for mixing with water and pressing the dried and ground raw material into bricks, which are filled into the kiln with alternate layers of coke, as previously described.

The new working of the kiln however entails a system of working these continuous shaft kilns under either a forced or induced draught. Both methods are employed in this country, but the forced draught arrangement which was introduced into this country by the author in the year 1906 has up to the present been found preferable. The arrangement involves the introduction of forced draught into each kiln under a pressure of about 4 inches W.G. at four equidistant points in the periphery of the kiln, all well below the firing zone and above the clinker drawing eyes, and where the forced draught must not be allowed to escape. By a series of four pipes of about 10 inches in diameter placed in equal spaces and having outlets on the inside lining of the kiln—all connected with a blast fan arranged in capacities to deal with the number of kilns to be treated—the output of the shaft kiln by the new forced draught arrangement is increased to double the usual amount, namely, up to 150 tons of clinker per week, with night and day continuous working, while the fuel consumption is reduced—say to 20 per cent.—and the quality of the cement is improved. Somewhat similar results are obtained by a method of induced draught with shaft kilns, in which case a suction fan is employed to draw the gases to the top of the kiln.

The **Stein** kiln consists of a vertical, parallel tube about 40 feet in height, made up of a number of concentric cast-iron rings, laid one on the other, the inside surface of each ring being smooth and the outside being formed so that when the rings are placed in position a series of vertical gills results which has the effect of keeping the rings cool, and preventing them from, burning or melting. The bottom of the kiln is fitted with bars in such a way that the kiln could be drawn when desired and yet kept sufficiently open to allow the necessary draught to pass. Doors are fitted to enable the bottom of the kiln to be made

air-tight in order to use forced draught of a pressure equal to 4 inches water gauge if desired, in which case the air would be supplied by a special fan.

A chimney some four feet in diameter and ten feet high is fixed to draw away the gases from the top of the kiln.

The Stein kiln is not used in this country, and is also of little use on a wet process works except where there is plenty of dry slurry to spare, or where it is dried especially for the purpose, but the kiln is very economically worked in connection with the dry process, burning dry or even damp bricks.

The clinker is drawn at intervals of from four to six hours, and the kiln is loaded in alternate charges of ordinary gas-coke and bricks, the usual charge of the latter being 20 to 22 cwts. of dry raw material. These charges are put on by hand from a stage on the loading level carried by girders and framing, and measuring about 16 to 18 feet by 11 feet, the kiln itself being a little less than 8 feet internal diameter.

The output from each kiln is from 150 to 170 tons of clinker per week of 7 days and nights, and the coke consumption is approximately 20 to 25 per cent. of clinker burned. The labour cost varies naturally according to the proximity and convenience of getting the dry slip to the kiln, but under ordinary conditions as to convenience, the cost for working this kiln is considerably less than with the older Chamber kilns.

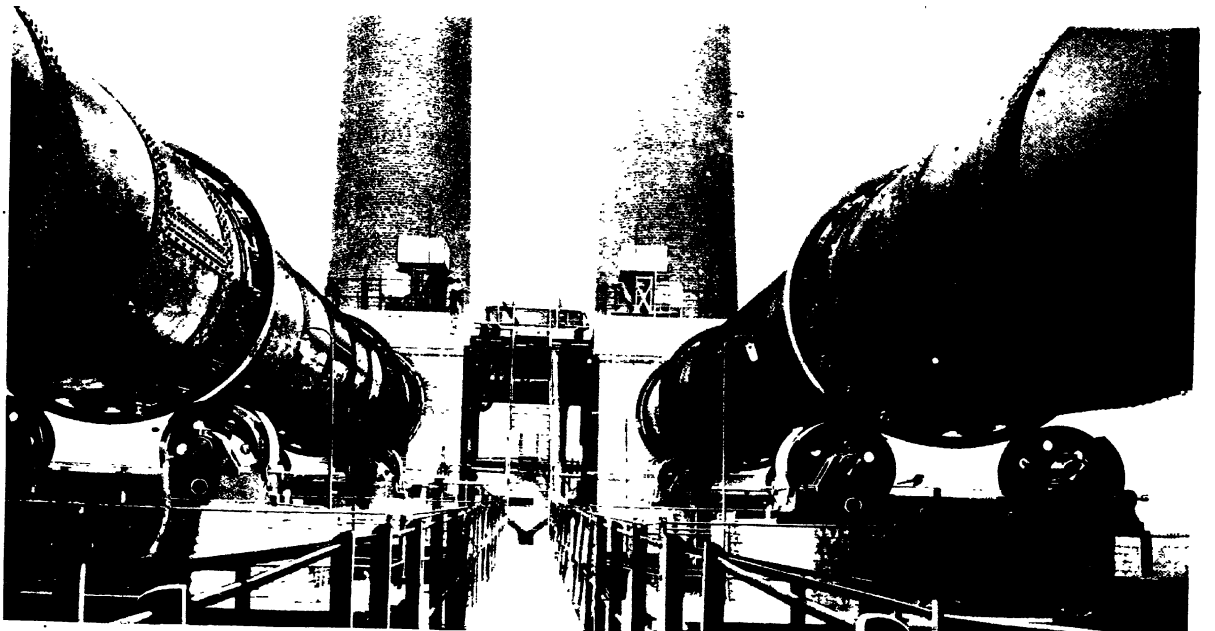
All the continuous kilns above-mentioned are mainly applicable to the dry process, but they can be used for wet process raw materials, and in this case supplementary drying arrangements are necessary by which the slurry may be dried sufficiently to allow of its being moulded into blocks. The amount of fuel required for drying is lower than that needed for burning, so that the total amount of fuel for both operations—viz., 30 per cent. to 35 per cent. calculated on the clinker—is not much smaller than that needed for chamber kilns, but a cheaper kind of fuel can be used, namely, small coal. In the dry process, however, the continuous kilns are always adopted with advantage.

The principle of continuous working is so attractive, on account of its economy in fuel and its increase of output per kiln, that it has been embodied in various other forms of kiln. These are, in general, vertical cylindrical shafts

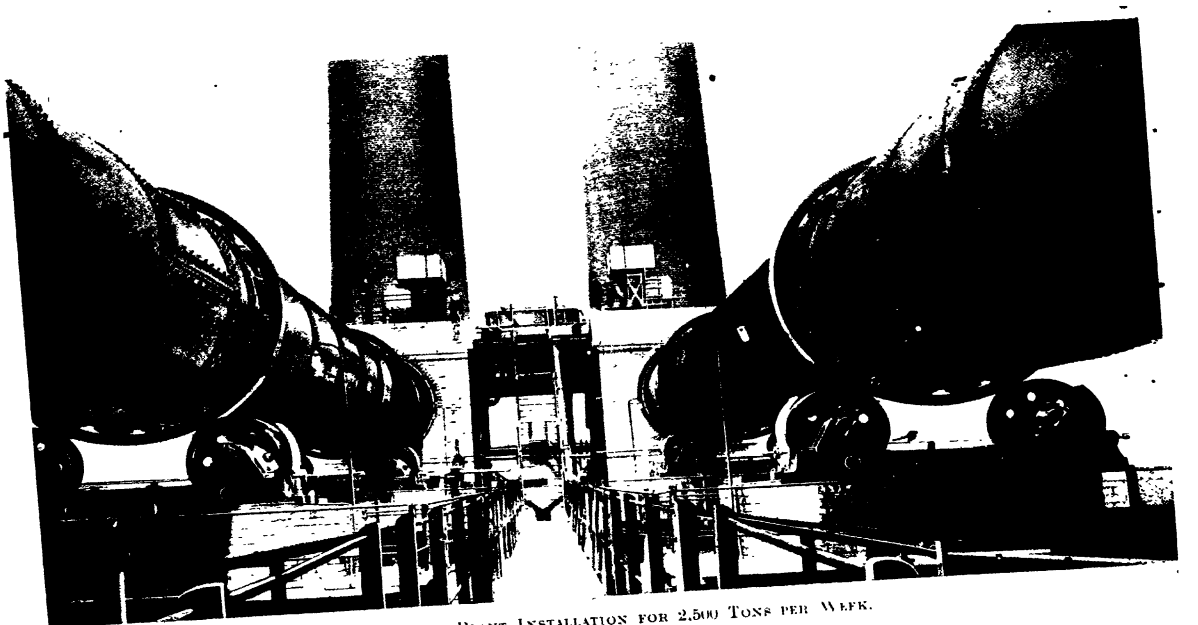
not unlike a running lime-kiln, but usually with a flue or chimney at the upper end, and loading doors at the base of the chimney. The clinker is drawn at the bottom, where there is usually some kind of removable grate.

Continuous kilns need some skill in loading. The hot plastic clinker is often liable to stick to the walls of the kiln, causing the charge to "hang up," and this not only hinders the proper working of the kiln, but also acts on the lining and hastens its destruction. Unless carefully worked, these kilns are apt to yield a good deal of underburnt clinker which has to be picked out before the clinker is ground.

The quantity of clinker produced by the different classes of fixed kiln now described varies considerably with local circumstances, but this may be stated in round figures as follows:—Dome, Batchelor, and Chamber kilns, 35 tons per week; Dietzsch kiln, 70 tons per week; Schneider kiln, 90 tons per week; forced draught shaft kiln and Stein kilns, 150 tons per week; and the Hoffman kiln, 28 tons per week for each chamber. With all these varieties of fixed kiln, whether continuous or non-continuous, the amount of labour needed for handling the raw material and clinker is larger than in the case of the Rotary kiln. Further, in whatever class of fixed kiln it is produced the clinker is somewhat irregular in quality and often contains imperfectly burnt material, and unless this is picked out with great care, sufficient underburnt material remains to impair the quality of the finished cement.



Rotary Plant Installation for 2,500 Tons per Week.



ROTARY PLANT INSTALLATION FOR 2,500 TONS PER WEEK.

The Rotary Kiln

CHAPTER VIII.

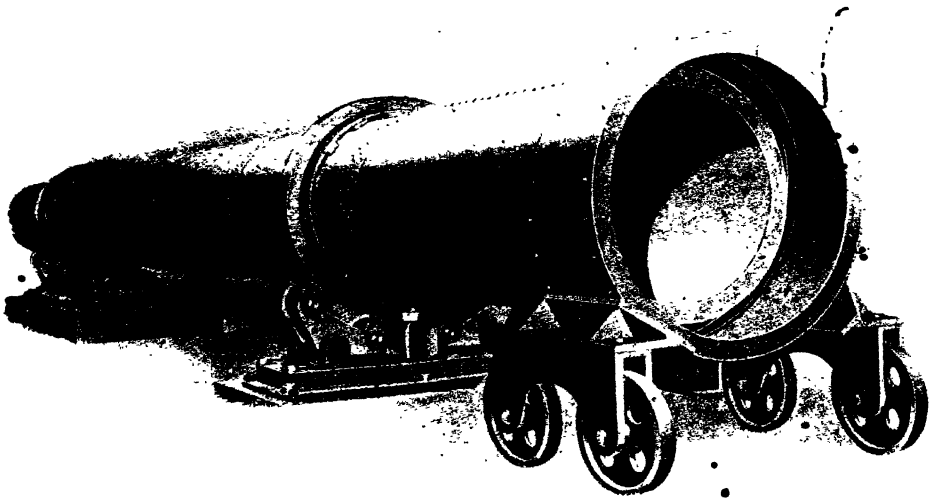
THE **Rotary Kiln** of to-day consists of a slightly inclined cylindrical tube in steel plates some ⅜-inch thick or even up to ¾-inch. Its length varies from 100 ft. to 250 ft., and in diameter the cylinder measures from 6 ft. 6 in. to 12 ft., according to the output required from the kiln—*i.e.*, a kiln of, say, 200 ft. long having a diameter of 10 ft. in the burning zone and 8 ft. 6 in. in the remaining length; one of 230 ft. length with 10 ft. diameter burning zone and 9 ft. in the remaining length; and a kiln of 160 ft. having a burning zone of 9 ft. diameter with 8 ft. for the remainder. These kilns have outputs respectively of 140, 180 and 110 cwts. per hour.

These diameter dimensions are exclusive of the usual 9 inches thickness of firebricks in the burning zone and 6 inches in the remainder of the kilns.

The long cylinder is inclined to the horizontal about 1 in 25 or 30 and is mounted by four or five sets of tyres (according to length), which run on heavy roller bearings and the kiln is slowly rotated by a train of gear wheels at a speed of 1 to 2½ revolutions per minute.

At the points of the kiln where the tyres and gearing are fitted the tube is re-inforced by additional plating.

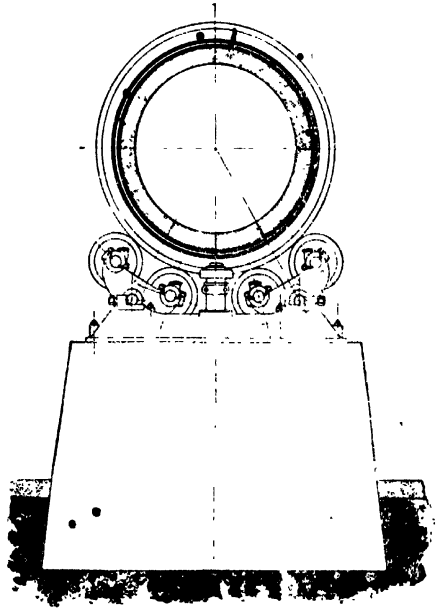
The cement-making materials are continuously fed into the kiln through a pipe at the upper end in the form of either slurry or dry powder, according to the process adopted in preparing and mixing the raw materials, which materials move by gravity from one end of the kiln to the other. Slurry lifters are fitted inside the kiln to spread the raw materials on entering the kiln and when meeting the hot gases. Finely ground coal is almost always used as



ORIGINAL TYPE ROTARY KILN SHELL AND HOOD BEFORE LINING.

fuel, and this is introduced into the lower or outlet end of the kiln by a jet of air issuing from a blast fan.

When the kiln is started the fine coal is ignited, and a white heat is obtained in the lower end of the cylinder. The raw material is then fed into the kiln, and as it gradually descends into the zone of heat generated by the perfect



ROTARY KILN.—Sectional View showing Carrying Mechanism.

combustion of the finely ground coal fed into the cylinder from the opposite end, it parts with any water that may be present, becomes heated to redness as it approaches the centre of the cylinder, loses its carbonic acid, forms little rounded balls which reach a nearly white heat in the lower end, and finally issues as well-burned clinker in grains about the size of a large pea. The greatest heat is naturally near the fuel jet or outlet end of the kiln.

The operation of calcining is a continuous one, and with proper care under or over-burning may be avoided. The hot clinker from the kiln is cooled in cooling towers or rotary cooling drums, but more usually in the latter.

The **cooling drums** now generally in use are placed at the lower end of the kilns, and receive the hot clinker as it drops from the kiln.

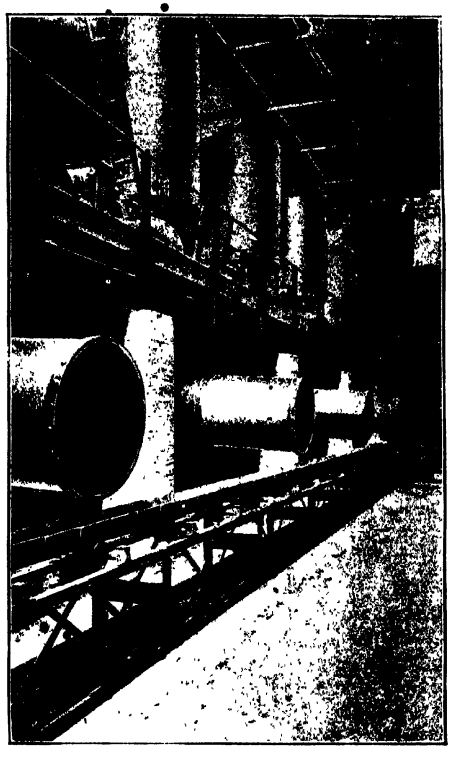
These generally consist of tubes some 40 to 60 feet long and 4 to 5 feet in diameter, but vary in dimensions according to the quantity of clinker to be dealt with. Inside each drum are cascading channels for lifting and dropping the hot clinker as the coolers rotate, thus presenting the clinker to the cold air forced through the cooler by a fan provided for the purpose. The coolers rotate at a somewhat higher speed than the furnace tube, and by the air for the latter being drawn through the red hot clinker a fair proportion of the otherwise lost heat is retained and utilised. When the clinker issues from the coolers it is quite cold enough to handle and to pass to the further process of grinding into powder.

The Rotary Kilns are not substantially economical in **fuel,** as they require some 28 to 35 lbs. of coal to produce 100 lbs. of clinker, and the greatest problem that confronts the manufacturer is the devising of some means for cheaper burning. The price of coke, however—the fuel used in the old-fashioned kilns to the extent of 8 to 10 cwts. of coke to a ton of cement—has advanced so much that the cost of cement production, by the older methods, has much increased, and further, the supply of coke is so limited and its utility has become so much greater in other directions that the Rotary Kiln has an immense advantage in being independent of a fuel which has now thus attained fictitious prices.

In the matter of **labour,** the Rotary Kiln is decidedly economical. The feeding of the kiln with raw material and fuel, being automatic, and no labour in "drawing" the clinker being necessary, a saving is effected in the cost of the burning process. Thus, the Rotary Kiln marks and, indeed, may be said to have created, a period of distinct development in the production of Portland cement.

All the earlier attempts in this country to construct satisfactory rotary kilns resulted in failure mainly on account

of the great difficulty experienced in obtaining a satisfactory **lining** for the furnace. Ordinary firebricks, being highly silicious or acid in character, united with the highly



BATTERY OF ROTARY CLINKER COOLERS.

basic clinker to form a fusible slag, and the lining was soon destroyed. Magnesia bricks were too expensive, whilst some of bauxite, tried by the original designers of the kiln, proved weak and friable.

In the early stages of the rotary kiln another difficulty arose in the matter of fuel. With producer gas it proved

difficult to obtain the requisite temperature in the absence of a regenerator, whilst crude oil, though successfully used in the States, was much too expensive to be thought of in this country. Most of the initial difficulties have, however, now been overcome by time and experience, and ground coal is usually adopted as the fuel for this kiln.

When starting the rotary kiln it is usual to form a protective coating of the clinker itself over the lining of ordinary firebricks. To this end the firebrick lining has in some cases been first covered with a layer of salt, and after raising the temperature of the furnace to the clinkering point, a charge of clinker is admitted. This reacts with the salt and silica of the firebrick, a glass being formed which welds the clinker and brick together, and the former is then well beaten into place. In this way it has been found that firebrick lining will last for a year though it is necessary to repair the clinker coating from time to time while the kiln is running. When the rotary kiln is stopped for any purpose this coating invariably falls off upon cooling and the same operation (with or without the use of salt) proceeds each time the kiln is relighted.

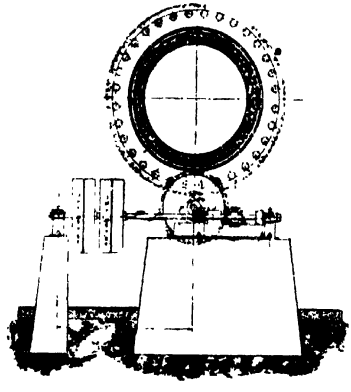
The **firebricks** used for lining the rotary kiln should be rich in alumina. A suitable block will contain some 60 per cent. of silica and 30 to 35 per cent. of alumina, the balance consisting of small quantities of impurities—viz., oxide of iron, lime, magnesia, potash and soda.

Fire blocks approaching this composition can be procured from several British firms, and makers of repute are generally willing to submit analyses of their goods.

The practice of providing linings to kilns with unfired clay blocks is sometimes indulged in. Fireclays, however, undergo on firing a certain contraction—maybe up to one-sixth—the actual contraction varying from clay to clay, and uniformity in mixing and burning firebricks is essential. It is obviously impossible to burn firebricks uniformly if they are fired *in situ* in the furnace where they are to be used and the hot face must contract much more than the parts nearer the external shell where the materials are cooler. As a result great strains must be set up in the brick, and the material does not get a chance to do its best work.

In the case more particularly of rotary kiln linings where a stoppage of the kiln is a serious factor, the firebricks used in the hotter portions of the cylinder should be tested for contraction, so as to make sure that the bricks in burning, or as supplied by the manufacturers, have had all the contraction taken out of them. If not, the bricks are liable to contract when in use, causing them to become loose and fall out of their position.

In cases where trouble arises at the hot end of the kiln



ROTARY KILN.—SECTIONAL VIEW SHOWING DRIVING MECHANISM.

with the bricks spalling and breaking, it is arranged that a more open textured brick is used. Close textured bricks are liable to split and spall with abrupt changes of temperature under conditions where more open textured bricks withstand the changes of temperature satisfactorily, and care should be exercised that the open texture is obtained by coarser or a greater proportion of what is technically termed "grog" or matrix, and not by the easy firing of the brick by the manufacturer. "Grog" is the coarse pre calcined material that is mixed with the clay in firebrick manufacture to prevent excessive contraction on firing.

In this direction a useful specification for rotary kiln lining blocks is as follows :—

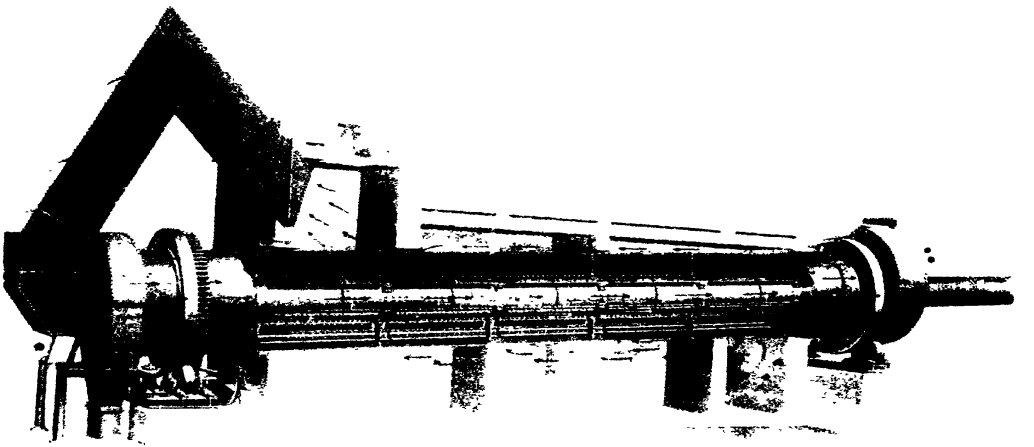
> A test piece, when heated to a temperature of Seger cone 16 for two hours, shall not show, when cold, more than 1 per cent. contraction or expansion. After the test temperature has been obtained, the furnace shall be maintained at a constant temperature throughout the testing period.

All rotary kiln plants that are fired with powdered coal must include an apparatus for the drying of coal to be used in the manufacture and also grinding plant for the same to be finely pulverised before entering the kiln. The most common design of **coal dryer** consists of an inclined steel cylinder encased in brick work. The hot gases from the fires coming first in contact with the cylinder at the point where the wet coal is introduced, pass along and around the tube on the way to the chimney stack, which is located over the discharge end.

In some cases the hot gases pass to another chimney whilst a separate chimney at the feed end of the rotary coal dryer permits the steam from the moist coal to escape and gives an outlet for any congregating gases or coal dust. In many modern installations, the coal is dried by hot air taken from the clinker cooler instead of by an independent furnace.

From the dryer the coal falls into a conveyor and is carried automatically to the **coal grinding mill.** This mill contains machinery for reducing the slack coal to an impalpable flour, and the plant commonly used is the ball and tube mill or the " Griffin " mill, illustrated elsewhere. This latter mill gives an output of some 1½ tons per hour to a fineness of not more than 1 to 2 per cent. residue on a 100 by 100 mesh sieve.

From the coal grinding mills the finely powdered coal is automatically conveyed to the rotary kiln house, where it is then elevated and again conveyed to the coal hoppers situated in front of each rotary kiln. The small store of coal in each hopper is sufficient to last the kiln for a few hours, and from this hopper an automatic feed conveys the powdered coal to a point where it is met by the heated air blast from the coolers and through the fan to the kiln.



Double Drum Rotary Coal Dryer

When reaching the hot zone of the kiln the powdered coal immediately bursts into flame and the intense heat is thus kept continuously regular.

A matter causing some difficulty in the early days of rotary kiln cement manufacture was in the **fineness** of the **coal dust** used for heating the kiln. It has been explained that this coal dust is blown in a fine stream into one end of the kiln by means of a blast of air, and the mixture of air and coal burns, under ideal conditions, very much the same as a mixture of gas and air would do.

Unfortunately, in the early days the coal used was far too coarse and much heat was lost on this account, to say nothing of the partially burnt coal which found its way into the cement and affected its quality.

When it is remembered that from 25 to 36 per cent. of fuel is needed to burn cement according to whether the dry or wet process is used, it will easily be understood that the question of using the fuel economically is a very important one.

It has been found that by using coal which left a residue of 15 to 18 per cent. of its weight on a sieve with 75 holes per linear inch, and 40 per cent. residue on a sieve with 100 holes, not more than 40 per cent. of such fuel would be available in the burning, even when working by the dry process—a loss of more than half the fuel consumed.

It has been seen further on examination that the coarse particles of fuel were difficult to ignite, and after glowing brightly for a moment they fell in a half-burnt condition amongst the clinker, and added greatly to the risk of an incompleted burnt product, as well as making it less pure. The ground coal used for firing should if possible not exceed a residue of 15 per cent. on a 180 x 180 mesh sieve, or even less if the coal contains a low proportion of volatile matter.

There is considerable danger in coal grinding on account of the explosive nature of fine coal dust, and caution is therefore necessary in preventing an escape of the very fine particles of coal. No naked lights must be taken near the coal grinding building. It is also on account of this explosive tendency and the risk of spontaneous combustion that the storage of ground coal is avoided as much as possible.

The **length** of the rotary kiln and its **output** at varying lengths is a subject now being studied by manu-

facturers, but it is, however, generally considered that according to present experience the smallest kiln to be used on the wet process shows more satisfactory working with a length of 120 feet and an external diameter of 8 feet, and the output from a kiln of this description is about 80 tons per 24 hours. On the dry process the smallest size usually employed is 80 to 100 feet in length and 7 feet 6 inches in outside diameter with an output of some 50 to 60 tons per day of 24 hours.

The capacity of a Rotary kiln is in the main governed by its size, but variations in the working of kilns of the same dimensions may be caused by a difference in the raw materials employed, in the angle of inclination of the kiln, the method of firing, the quality and fineness of fuel, the percentage of moisture in the raw material, the skill of the attendant burners and freedom from stoppages.

Some interesting information upon the variation in the output of Rotary Kilns at different lengths has recently been published by the United States Geological Survey Department from statistical data received from various cement plants. By means of these data which cover a very wide range of material, practice, and operating conditions, it seems probable that average results of fair accuracy can be deduced as to the effect of raw material and length of kiln on the output. This has, at any rate, been attempted, and the results are presented in the following table. In explanation of this table it may be said that the average annual output of the old 60 ft. Rotary kilns, fired with powdered coal and working on a dry mixture of limestone and clay, is taken at 100 per cent. The average outputs of similar and longer kilns working on various raw materials are then compared with this standard, the results being expressed in percentages.

PERCENTAGE OF VARIATION IN OUTPUT WITH LENGTH OF KILN, RAW MATERIALS, ETC.

Raw Materials	Length of Kiln				
	60 ft.	80 ft.	90 ft.	110 ft.	150 ft.
	%	%	%	%	%
Low carbonate limestone	126	164	182	216	400
Limestone and clay ...	100	150	166	190	350
Chalk and clay (wet process)	54	76	95	105	250

As to the **angle of inclination** it has been shown by experience that 1 in 30 gives the best results. The greater the incline the quicker will the feed approach the outlet end of the kiln, and in some cases, such as with a kiln inclined to 1 in 15, the raw material fed lurches forward to the clinkering zone immediately it is fed to the kiln. This necessitates the continued stopping of the kiln to burn the material back from the outlet end and the clinker is of variable quality.

In the case of a kiln inclined at the lesser angle, the feed to the firing zone is regular and continuous, with the result that the raw materials are slowly brought into contact with the hot gases with a consequent increase of output and fuel economy and the manufacture of a more regular product.

The present tendency towards the lengthening of short rotary kilns for cement burning to lengths of 150, 200 and even 250 feet, is indicative of the desire on the part of the cement manufacturer to gain greater fuel economy and output, even though the changes require the complete abolition of his kiln room, and the construction of practically a new cement burning equipment, all at great expense; but recent experience shows that the results of the trials with the extremely lengthened kilns have not been so satisfactory as may have been anticipated.

The incorrectness of predictions with regard to the length of kilns seems to be due in a large measure to the failure to consider a further important factor in rotary cement burning, namely, the quality of the calcining **flame.**

The rotary cement kiln has to perform two operations which are essentially quite distinct chemically and otherwise one from the other. In the upper part of the kiln efficient calcination should take place, that is, the carbon dioxide contained in the chalk or limestone should be well driven off in order to fit the material for the subsequent changes which it has to undergo in the clinkering zone. Calcining calls for a relatively low temperature as compared with clinkering, and necessitates a heat which in steel furnace practice is termed a "soaking" heat, by which is meant a reducing or non-oxidising flame. In steel furnace practice such a flame is often desired to prevent oxidation of the charge. The same result, however,

is not obtained in a rotary kiln, since the flame operates by its heat alone, and not by chemical action.

It is apparent therefore that a small volume of intensely heated gases, or a larger volume of gas at a lower temperature may be discharged into the mouth of a rotary kiln, and effect quite different results. The latter effect is said to be produced when a mixture of air and stack gas is used for combustion instead of pure air. It is maintained further that it is only by having a flame peculiarly described as a "reducing" or "non-oxidising" that calcination can be made rapid and thorough.

With the usual methods of burning cement by employing powdered coal and ordinary air, the necessary flame is often not properly produced in the region of the kiln devoted to calcination. With longer kilns the difficulty is much aggravated, and to this imperfect flame development the failure of the long kiln to fulfil the prophecies of its promoters is attributed. Inventors, however, have been busy in this field of research, and already certain methods have been put to a practical use which permit of a betterment of operation. Of these, perhaps the most promising is the system of the **impinging flame** in cement burning.

Some of the advantages to be derived from burning cement clinker with a flame having a positive impinging action on the material and the economy to be gained by the use of the impinging flame instead of the axial flame (or flame substantially parallel to the axis of the kiln, which does not impinge upon the material and heats the latter by radiation solely) have been much discussed in America, and in order to better understand the application of flames of the character above-mentioned, reference has been made to the methods employed in two of the great industries of that country, namely, the manufacture of steel and glass.

From deductions made from experience in these industries, it is maintained by the advocates of the impinging flame that in burning lime and cement, in annealing and heating steel, and in many other industrial processes involving higher temperature, the methods of combustion best suited to each operation require modification. In such processes air contains too much oxygen for the kind of flame required. A number of attempts to

produce a flame of low temperature and large volume have been made in the past, such as those using steam as a diluent, but they have proved either impracticable or uneconomical. It has frequently been proposed to return part or all of the gases of combustion to the kiln in order to save in some way the heat these gases contain, but the suggestion has not been practically successful on account of the great volume of the returned gases, and the lack of comprehension of their true effect on the fire.

This principle was substantially as follows:—A flue is tapped into the stack of the kiln high enough to secure gases of fairly uniform quality which are led to a fan. At one portion of its length there is an air inlet with an adjustable damper at its mouth. The fan has two outlets, one under the grate and the other into the open air. This arrangement makes it possible to draw air and gases through the fan in any desired proportion by adjusting the air inlet valve. By controlling the damper of the free discharge opening and changing the speed of the fan, the pressure and volume of the blast under the grate can be modified through a wide range. When the apparatus is properly adjusted the combined effect of the blast and the diluent is to evolve the heat of the fuel where it is most needed, instead of at the portion of the kiln where it occurs under natural conditions. The temperature of the furnace is thus lowered and that of the charge is more uniformly distributed than with the pure air draught. As a result of this arrangement it has been claimed that the output of an ordinary kiln is raised, while the amount of fuel is reduced and the quality of the cement is unchanged.

While the rotary kiln is acknowledged to be wasteful in fuel, this defect is more than counterbalanced by its large output under skilful supervision.

The powdered coal blown into the kiln burns with an intense heat, and if the flame deflects much from an axial line, there is a tendency for the material to form rings and even for the lining to deteriorate very rapidly Consequently the region of maximum heat is along the centre line of kiln, and not direct on the material at the clinkering zone.

With an impinging flame it becomes possible to produce such a temperature that the flame can be directed im-

mediately on the material which will have its maximum temperature in the clinkering zone.

With the modern Rotary Kiln now generally brought into use in the manufacture of cement, the industry has been revolutionised and its methods are at present perhaps the most scientific and practically efficient that have been introduced in the manufacture since Portland cement was first known. This kiln is, of course, now in general use in this and every other country, and it may be said that the Rotary Kiln is the only invention of importance in the manufacture since the introduction of the ball and tube mills for grinding. It may be expected, therefore, that a decade at least will elapse before any further development will arise of such an equally important character. That such improvements will occur, however, is quite possible and indeed probable, for even with the use of the Rotary Kiln the manufacture of Portland cement is a tedious, technical and expensive process involving heavy capital outlay, high cost of production and continuous upkeep, and with the present kilns there is certainly an immediate demand for fuel economy, to say nothing of minor improvements which will no doubt be introduced from time to time as experience provides the necessity and time gives the opportunity.

More than ninety per cent. of the cement manufactured in the States is produced by the Rotary Kiln, and in England there are many works—if not the bulk of them—in which this Kiln is solely adopted. Its use in all directions is also increasing, and many obsolete factories, not only in this country but in many parts of the Continent, are now installing the Rotary Kiln for cement burning and most new works adopt this process of burning.

Although quite a comparatively new method of cement manufacture, enough has been seen of rotary cement, both in science and practice, to declare it to be the product of the future.

To advocate the use of Rotary cement is unnecessary, since all that science can bring to prove its quality, and all that practice can offer to show its superiority so effectually establishes its claim for preference that we may say without fear of contradiction, Rotary cement now leads the way.

The product from the Rotary Kiln has a distinct ad-

vantage in quality both for high tensile strength with age and absolute freedom from all expansion and contraction tendencies. This improvement is only to be expected, however, for apart from its economy in working, the great



Old Type Rotary Kiln Ready for Firing.

advantage of this kiln is that the process of calcination is in full view of the operator, and can be controlled by him to a nicety. By altering the rotating speed of the cylinder, and decreasing or increasing the feed of the raw mixture or varying the force of the coal blast and the quantity of

fuel the burner is, or ought to be, able to regulate the operation of burning to any desired degree.

With the old-fashioned intermittent kiln no such adjunct is possible, for when once the kiln is charged and fired the calcining proceeds automatically for better or worse, and the product from the kiln must take its chance.

After the clinker from the Rotary Kiln is ground it is generally found to be of an extremely **quick-setting** nature (owing to the absence of sulphates mainly derived from the coke or other solid fuel generally used with other processes), and arrangements have to be made by the manufacturer to overcome this difficulty, such as by aerating or hydrating the clinker before grinding, or again, by the introduction of steam into the clinker grinding mills or by the addition of a small percentage of gypsum or plaster of Paris to the clinker.

The great simplification of working which is produced by the use of Rotary Kilns in place of the older shaft kilns in the burning of cement is, however, perhaps more apparent than real, because the working of the Rotary Kiln, though theoretically very simple, is in practice somewhat complicated.

For instance, the size of the particles of the raw materials is of equal importance to the proportions in which they are used, and it involves the employment of skilled workmen, with equal, if not greater, intelligence than that required for the older form of kiln.

The product of the rotary kiln is purer than that of other kilns because in the latter, the ash of the fuel is mixed with the clinker and is ground as cement, while in the rotary kiln the greater part of the fuel ash is blown through the kiln by the force of the fan blast.

It occasionally happens however that with coarsely ground coal containing a large proportion of ash, so much of the ash is deposited on the lining of the kiln burning zone as to form "a ring," and unless the use of the defective fuel is stopped, the "ring" attains such proportions as to render burning impossible, and then the kiln must be stopped while men go inside to free the lining.

An occasional rapid loss in the strength of Rotary Kiln cement on keeping is not infrequently due to the use of too coarse a coal as fuel, or the admission of too little air for its complete combustion. In either case the formation of

carbon monoxide results in a partially burned cement being produced.



Modern Rotary Kiln and Double Shell Cooler with Clinker Conveyor.

The best preventive of this is the frequent testing of the flue gases whereby the correctness, or otherwise, of their composition may be ascertained, and, if necessary, an alteration in the supply of air effected.

The Rotary Kiln. Fuel Valuation.

CHAPTER IX.

THE quality of **coal** best suited for use with the modern rotary kiln is a bituminous slack, containing not more than ten per cent. of ash, five per cent. of moisture, and one and a half per cent. of sulphur. It should have a heat value of not less than 7000 calories per kilo, which is equal to 12,600 British thermal units per lb. of coal and the proportion of moisture and ash should be kept as low as possible. The average results of rotary kiln coal analyses in bulk as delivered at works are given below.

These are of fair quality slack coals from the collieries of the Midlands in England.

Analyses of average Bituminous Nutty Slack Coals from the Midlands (England).

Sulphur	Water	Volatile Matter	Non-Volatile Carbonaceous Matter	Ash	Calories per kilo of Coal	B.T.Us. per lb. of Coal	Lbs. Water evaporated at 212°
%	%	%	%	%			
1.98	6.27	36.98	51.27	5.48	6830	12294	12.74
1.50	6.79	37.07	47.96	8.18	6737	12127	12.57
0.65	8.66	37.42	47.13	6.79	6514	11725	12.15
1.00	9.59	35.56	46.45	8.40	6302	11344	11.76
1.60	5.4	34.9	50.1	9.6	6435	11608	12.0
2.3	5.6	34.2	49.0	11.2	6160	11088	11.4
1.0	5.4	31.6	55.9	7.1	6930	12496	12.9
2.0	6.8	35.8	48.5	8.9	6765	12202	12.6
1.56	7.3	40.1	46.6	6.0	6710	12110	12.5
1.7	5.8	32 0	58.0	4.2	7260	13068	13.5

The **combustion** of coal is a chemical process, and its behaviour during combustion is a physical phenomenon. Each must be considered when the aim is to extract the maximum number of units of heat from a given weight of fuel, for either a variation in the chemical characteristics, or an improper control of the kiln is liable to lead to deceptive conclusions.

In order to obtain accurate information in regard to the number of heat units which complete combustion of the fuel will yield, one must resort to chemical analysis or to the calorimeter test, either of which, if properly carried out, postulates the selection of suitable fuel for the work in hand, and, what is often of greater importance, should really determine the exact basis of payment between the consumer and the supplier.

The practice of selecting fuel by simply inspecting samples of the kind of coal which the merchant agrees to deliver is irrational to the last degree, for no one can obtain definite knowledge of the heating value of any coal by even the most critical optical examination. Nor is it possible to decide fairly, by such a method, between two coals of about the same quality, but differing slightly in appearance. Nothing short of the work of the laboratory can solve the problem.

For use with a rotary cement-making plant, the **selection** of coal is almost as important proportionately as is the choice of raw material for a factory. The calorific power is frequently but one of several factors to be considered. Although the heating value of a unit of carbon or of hydrogen in all coals of the same nature is the same, there is no doubt that in some cases the adaptability of a fuel to the particular furnace and service in hand is of sufficient importance to warrant its acceptance or rejection, quite apart from its relative heating power in comparison with that of its competitors.

The demand of the manufacturers for coal of uniform quality is reasonable, but in the exigencies of practical mining it is difficult perfectly to sort out inferior grades and partings of slates, and in actual practice the fact that a coal bears a certain name is really of little significance as to its quality.

Within reasonable limits it is now possible to specify and obtain coal of practically uniform quality, but in any event

the chemical analysis and the calorimeter test enable the purchaser to obtain his money's worth of heating power.

One of the most important phases of the coal testing problem is the impartial **selection of samples.** The personal equation must be eliminated in the effort to obtain a fair average of the barge load of truck load or heap.

Samples should be taken from a number of different places in the stock equidistant from one another, including large and small lumps, regardless of different quality. These samples should then be united and crushed, mixed, and sub-divided until quartered selections of 20 or 25 lbs. are obtained. These should be kept secure for the purpose of analysis. The final samples for the chemist should be powdered coal capable of passing through an 80 to 100 mesh screen; these should be kept in tightly stoppered glass bottles. It is remarkable how little discrepancy will often be found between properly-selected samples from the same barge load. But unless impartial sampling is carried out, the most painstaking work of the laboratory is of little avail in establishing the correct conclusions.

In urging the influence of **coal testing** upon industrial economy, a few points may be noted in regard to the interpretation of chemical analysis. The literature of this subject is full of formulæ for the heating power in terms of the composition, and in many cases equations have been derived from the quality of coal found in particular localities. The ultimate analyses give results which generally check far closer with calorimetric determinations than does the proximate analysis, and the latter should not be used alone when an accuracy within narrower limits than 2 or 3 per cent. is desired. **Ultimate analysis** reduces the fuel to its elements, hydrogen, oxygen, nitrogen, sulphur and carbon, with ash and moisture. **Proximate analysis** determines the percentage of fixed carbon, volatile matter, moisture and ash.

Probably the most satisfactory determination of the calorific power of fuel to-day is that effected by the calorimeter, various apparatus being here explained, and these actually burn a sample of fuel under known conditions, and measure with great accuracy the rise in temperature of a surrounding water bath.

In the combustion required to produce the necessary heat in a rotary kiln for cement burning, many kinds of fuel

are used, including anthracite coal, but those giving an analysis on the lines of the results before mentioned are suitable. As explained, however, fuel contains chiefly carbon and hydrogen, and it is when these combine with the oxygen of the air that they give out heat. The problem is to generate as much heat as possible for each pound of fuel and to utilise it completely in the kiln, whether the heat for the rotary kiln be evolved from oil, gas or finely ground coal. The latter means are most generally adopted in the manufacture of cement by the rotary kiln in this country.

To obtain the maximum amount of heat, combustion must be complete. This involves compliance with certain conditions, not always easy to secure. In the first place, the requisite quantity of air must be supplied, and this amount varies with the particular fuel used, all coals differing in composition. Air is needed in order that the oxygen it contains may combine with the carbon and hydrogen in the fuel to produce combustion. The oxygen is present with nearly four times its weight of nitrogen, an inert gas which neither burns nor supports combustion, but which must, nevertheless, be heated up to the temperature of the flue gases, thus absorbing much of the heat generated by combustion. This loss of heat is unavoidable, but it can be reduced by carefully regulating the admission of air, and some of it is returned to the kiln through the clinker coolers.

The indraught about the ends of the rotary kiln can be used or prevented at will, and the point of highest temperature can be regulated by the burner in charge, who can focus it farther up or down the kiln as desired, and thus avoid burning out the lining in one place.

The heat from the clinker is generally used to heat the coal blast.

In rotary kilns fired with coal a better heating effect is obtained when the fire jet is directed slightly to one side of the kiln and downwards.

The jet should be mounted therefore so as to be capable of taking any radial position.

In all coal used for rotary kiln burning the heat value of the fuel should be constantly ascertained both before and after purchase. The heat of combustion of the different elementary bodies of which the fuel is composed

has long been known, and if the composition of the coal is determined by analysis, its heating value may be thence calculated by adding together the heating value of its constituents and allowing for their heat of combination.

The **calorific power** of fuel, for instance, is the number of heat-units obtained by the combustion of a unit weight of the fuel in oxygen. The laboratory determination of the calorific or heating value of a solid fuel is carried out by burning a weighed amount of the dried sample in oxygen in a closed vessel under water, and noting the increase of temperature that results from the heat generated by the combustion. If the weight of the water by which the vessel containing the burning sample is surrounded be known, and if the whole of the heat be abstracted from the gaseous products of combustion, the rise in temperature of the water multiplied by its weight gives the number of heat units developed by the burning of the fuel.

Two sets of units are in use—the **British thermal unit,** or B.Th.U., which represents the heat required to raise 1 lb. of water through 1 deg. F., and the **calorie, or metric unit,** which represents the heat necessary to raise one gramme of water through 1 deg. C. A larger metric unit based on the kilogramme is also in use.

If large quantities of fuel be employed in these determinations, the heat losses due both to the size of the apparatus required, and to radiation, are proportionately high and difficult to allow for. The chemist surmounts this difficulty by taking great care in the preparation of the sample used for the calorimeter, and by working with a small amount of fuel. The general practice is to use only one gramme of fuel for the calorimeter test: special precautions are therefore necessary to obtain the required degree of accuracy in the weight and temperature measurements. Some chemists prefer to work with 0.4 gramme, and 3,500 grammes of water. Under these conditions the rise in temperature of the water is usually less than 1 deg. C., and all corrections for radiation losses can be dispensed with. Very accurate and standardized thermometers, which can be read to 1-100th of a degree, are needed, however, when working with such small amounts of fuel.

Oxygen is used for the combustion, although air would be more in accordance with the conditions of practical work. The difficulties of igniting and burning a coal

sample in a current of air in a confined space without production of smoke and soot are so great, however, that oxygen has been generally accepted as the only possible substitute. Two methods of applying it are in use. Either the fuel sample is burned under normal atmospheric pressure in a calorimeter of the "submerged bell" type, or the fuel is burned under high pressure in the "bomb" type of calorimeter. In skilled hands either method gives reliable and concordant results, but as the decomposition products of coal vary in character with the temperature, the calorific values obtained by the bomb apparatus are sometimes higher than those obtained by the bell.

Heat Insulation.—In using either the bomb or the bell type of calorimeter it is essential to have some protection against temperature exchanges between the vessel holding the measured volume of water and the outside air. The most practical form of insulating vessel consists of an outer water-jacketed vessel containing a stirrer and enclosing an inner air space, in which the highly polished nickel-plated vessel containing the water is supported on three small heat-insulating corks. When in position the inner vessel is thus surrounded by an air jacket and by a water-jacket, and if a circular asbestos board or plate (with the necessary openings in it for the thermometer, electrical ignition wires, and oxygen-supply tubes) be employed to close the top of the vessel, the temperature exchanges with the outside air will be reduced to a minimum. Some form of mechanically operated stirrer is used before and at the end of the test to obtain a thorough mixture of the water in the inner vessel. Electrical ignition is often used with both types of calorimeter.

Briquetting the Samples.—A small briquette or tabloid mould often completes the apparatus required, since if it be attempted to burn the fuel in powdered form, low results will be obtained. This deficiency is due to portions of the fuel been blown upon the metal base of the bell when the ignition starts, and also to the ash which will form above and cover the lower layers of fuel as the combustion approaches completion. The necessity for pressing the fuel into small pellets or briquettes extends also to bomb tests, since if the fuel be in powdered form, some portion of it is thrown against the internal walls of the

bomb by the force of the explosion, and will there escape combustion.

Bituminous fuels as a rule will form pellets and briquettes by pressure alone. In cases where insufficient tarry matter is present in the natural fuel, just sufficient of a 1 per cent. solution of gum arabic may be used to make the particles of fuel adhesive. For half a gramme of fuel, three small drops of such a solution will suffice. The pellets or briquettes must be heated in the air-bath at 230 deg. F. (110 deg. C.) for at least four hours to expel the last traces of moisture thus introduced before testing in the calorimeter.

Standard Types.—Two standard examples of the bell and bomb types of calorimeter will now be described, but it must be understood that many different makes of these two types are in general use, and that practically all accurate calorimeters belong to one type or the other. Since accuracy in calorimetric work can be attained only by practice and by close attention to minute details which cannot be set out here, readers are referred to the various handbooks on this subject for more complete information as to the methods of observation.

The **Darling bell apparatus** consists of a glass bell-jar closed above by a rubber cork, and provided below with a glass flange ground quite level on its lower side. By the aid of rubber rings, a circular brass ring and small milled nuts and screws the glass bell can be firmly fastened down upon a brass support, through small holes in the base-plate of which the gases produced by the combustion of the fuel have to pass before they can escape and bubble up through the water. In order to break up the bubbles more completely, a piece of brass gauze may be fixed on the bell, or better still, a copper spiral may be provided above the brass base, and all the gases produced by the combustion may be forced to pass up this spiral before they escape through the water. Such a copper spiral is of great service in extracting the last traces of heat from the waste gases in all calorimeter work.

The **Mahler-Donkin bomb calorimeter** consists of a massive gun-metal cylinder, provided with three projecting thick screwed stud-pins for bolting down the cover. The

cover is provided with a milled-head screw valve for regulating the inlet of oxygen to the cavity inside the bomb. There is also an insulated conducting wire, which runs through the cover and terminates above the brass wire ring used for supporting the small platinum capsule or crucible containing the fuel. Thin lead wire is used to make a tight joint between the bomb proper and its cover, a circular groove in the cylinder top having as its counterpart a projecting ring on the under side of the cover. The bomb is tested up to 1,800 lb. pressure before it is sent out, and is plated inside with gold, in order to withstand the action of the nitric and sulphuric acids produced by the combustion of the fuel.

The usual pressure used in these tests is 20 to 27 atmospheres, or 300 lb. to 400 lb. It is advisable, therefore, to have a small back-pressure valve inserted in the milled head-screw in the bomb cover in order to avoid a great loss of gas when disconnecting the oxygen supply pipe and gauge from the bomb, after filling the latter with oxygen. At these high pressures the combustion of the coal is practically instantaneous, and the thin platinum wire used for ignition purposes will generally be found fused owing to the temperature momentarily attained. In order to protect the platinum capsule or crucible from the same effect and from the action of the molten slag produced, it is necessary to line it with thin asbestos board, cut and shaped to fit the crucible or capsule. This asbestos board must be dried and ignited before use in order to remove all matter that might vitiate the test results.

As regards the comparative advantages of these two types of calorimeter for regular routine-fuel-testing work, the bell apparatus is less costly to set up and maintain in working condition, and the glass bell, if carefully used, will outlast two or three bombs with gold or enamelled linings. The only durable type of bomb is that with a platinum lining, and at the present price of platinum the cost of such a bomb is prohibitive. Further advantages of the bell type are that the combustion is visible, it being possible to observe its progress through small holes cut in the cover of the calorimeter, and that being carried out under only a slight plus pressure it is more in accordance with the conditions obtaining in practical work

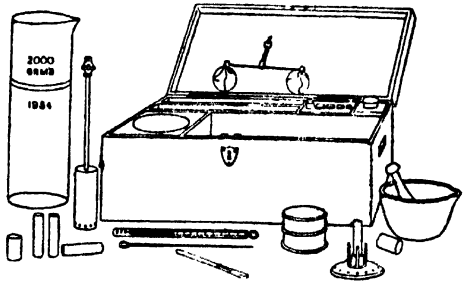
than the combustion in the bomb, which attains the violence and rapidity of an explosion. The rate of burning the briquettes or pellets in the bell can, in fact, be brought into exact ratio with the rate of combustion in the boiler furnace. A calorific value obtained under such conditions is of more practical value than one obtained by instantaneous combustion under 400 lb. to 500 lb. pressure, with the attainment of a temperature at which platinum melts. The decomposition products of coal vary considerably in composition with the temperature attained during their evolution, and it is reasonable to assume that the gaseous products evolved in the bomb under the conditions obtaining during a calorimeter test are quite different in composition and heat value from those evolved at much lower temperatures and under normal pressure. For scientific work the bomb is, no doubt, preferable to the bell, but for everyday use and for the practical requirements of large works the bell type has much in its favour.

The **Lewis Thompson's Calorimeter** here illustrated is another apparatus often used, and although the results by this method of testing are only approximate, they are quite sufficient for a commercial working valuation. This apparatus consists of a glass cylinder holding 2,000 c.c. of water, that is, 2,000 grammes when filled to this mark. The temperature of the water is taken with a delicate thermometer before beginning the experiment. Then two grammes of powdered coal are mixed with 11 grammes of the oxygen mixture (1 part potassium nitrate to 3 parts potassium chlorate, this being finely ground and well dried). A little of this coal mixture should first be tried to see if it burns properly. If the mixture does not burn steadily, the amount of oxygen mixture must be varied. When a mixing is found to burn well, a similar mixture is made and transferred to the narrow copper tube, the bottom of which should be tapped, while small portions are being pressed down gently with a test tube. When all the mixture is added, a piece of fuse about $\frac{1}{2}$ inch long is inserted : this fuse is made by soaking thin cotton wick in a solution of potassium nitrate and drying.

The tube is now placed in the clips of the brass bottom, the fuse lighted and the cover placed quickly over it, care

being taken that the stopcock is closed; the tube is immediately immersed in the water contained in the glass cylinder The cover consists of a copper cylinder with a narrow tube; this prevents the water from coming in contact with the mixture. The products of combustion escape by holes at the bottom, and the narrow tube is provided with a stopcock which is opened to admit the water into the cylinder and tube after the combustion is finished.

When the combustion is finished, the stopcock is opened to allow the water to rise into the cylinder. The apparatus is then raised and lowered several times to mix the water. It is then removed and the highest temperature attained



THE LEWIS THOMPSON'S CALORIMETER.

taken and the increase noted. The calculation is then made as follows:—As 2 grammes of coal were taken and the cylinder contained 2,000 grammes of water, the number of centigrade degrees increase in temperature multiplied by 1,000 will give the heat units evolved by the coal. The amount of heat absorbed by the apparatus during the combustion and so rendered insensible to the thermometer is equal to 10 per cent., therefore 10 per cent. must be added to the number of degrees of temperature which the water has been raised.

Example:—

Initial temperature of the water, say, 15.6°C., and the
Final temperature of the water, ,, 22.2°C.

The correction for the 10 per cent. absorbed is now added, thus :—$(6.6 + 0.66) \times 1{,}000 = 7{,}260$ calories, or heat units.

To convert the calories per kilo. into B.T.U. per lb. **(British Thermal Units)** multiply by 9 and divide by 5, thus :—

$$\frac{7260 \times 9}{5} = 13068 \text{ B.T.U. heating value of coal,}$$

the British Thermal Unit being the quantity of heat required to raise one pound of water one degree Fahrenheit.

As before explained, the ordinary **chemical analysis of coal** generally consists in the estimation of the following :—Moisture, ash, sulphur, volatile matter, non-volatile matter or coke, and the calorific power.

The results obtained will usually enable an opinion to be formed as to the quality of the coal.

If the coal is well sampled and ground to a fine powder, the requisite tests may be carried out as follows :—

Moisture.—Weigh out two grammes of coal in a watch glass and place in a steam oven. Remove the coal in about an hour's time, cool in desiccator and weigh. The weight decreases by loss of moisture, but after a time increases by oxidation, so a series of weighings are carried out and the lowest figure taken as the completion of drying.

Ash.—Carefully weigh out one gramme of the powdered coal in a shallow platinum dish ; this is placed on a nickel or pipe clay triangle and ignited slowly at first, but afterwards more strongly till only a clean ash remains. It is then cooled in a desiccator and weighed.

Volatile and Non-Volatile.—Weigh out one gramme of the powdered coal in a platinum crucible which has been previously weighed without the lid. Cover the crucible with the lid and place on a triangle and heat with a full bunsen flame. When the flame from the gases coming from under the lid stops, heat for about two minutes longer, remove the flame, cool in the desiccator, and weigh as soon as possible without the lid.

In order to have comparable results in different opera-

tions the size of the flame and position of the crucible must be the same in all estimations.

The loss in weight is the total volatile matter, and by subtracting the moisture already estimated, the actual volatile matter is obtained.

The residue in the crucible from above estimation is the coke containing the ash, so by subtracting the weight of ash from this, the non-volatile carbon is obtained.

Example :—

	Moisture found	5.6 per cent.	
	Ash found	7.3 „	
Volatile matter	Crucible + coal before heating		13.9630
	„ „ after „		13.6172
	Total volatile		.3458
	which equals		34.58%
	Less moisture		5.60%
	Actual volatile matter		28.98%
Non-volatile matter	Crucible—coke		13.6172
	Crucible alone		12.9630
	Non-volatile and ash		.6542
	which equals		65.42%
	Less ash		7.30%
	Actual non-volatile		58.12%

Sulphur.—Weigh out one gramme of coal in a platinum crucible, and thoroughly mix with about four times the weight of finely powdered dry sodium carbonate. The mixing is effected by adding the sodium carbonate gradually to the coal in the crucible, and stirring constantly with a dry glass rod.

Partially cover the crucible with the lid, and heat very gently at first, so as not to volatilise the hydro-carbons. Gradually raise the temperature nearly to redness, and continue the heating until the mixture becomes faint grey; now raise the temperature to a dull red heat for about an hour or until the mixture has become almost white, owing to the combustion of the coal.

Now treat the cool mass with water, digest on a water bath for a time, and filter the solution.

Add a few c.c. of bromine water to the filtrate, and acidify with hydrochloric acid; then boil the liquid until all the bromine is removed. Add barium chloride solution and boil; allow the precipitate of barium sulphate to settle; filter it off and wash well. Transfer the moist filter paper and precipitate to a platinum crucible, ignite, cool in desiccator and weigh as $BaSO_4$.

Calculate as follows:—

$BaSO_4 \times .1374 \times 100 =$ per cent. sulphur.

In this estimation the mixture must not be stirred during the heating, nor should any smoke or odorous gas escape. If much iron is present the mass will be of a red colour after heating.

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The Efficient use of Fuel in Rotary Kilns

CHAPTER X.

Of extreme importance to the cement manufacturer is the question of the efficient utilisation of heat when clinkering raw material in a Rotary Kiln. The problem is somewhat similar to one that has long exercised the attention of mechanical engineers, viz., the conversion of the greatest possible proportion of the heat set free in the burning of coal into steam in a boiler. An enquiry into the amount of heat dissipated and wasted in clinkering raw materials necessarily follows somewhat along the lines already laid down in investigating the efficiency of steam boilers, that is, in ascertaining the total heat given out in burning the quantity of coal used in making one ton of cement clinker, and comparing it with the heat which is theoretically necessary.

It will be convenient to consider separately the various operations in which heat is usefully expended, recovered, or wasted.

(*a*) **Heat is usefully expended in—**

(1) Driving off moisture from the raw materials.
(2) Heating up the raw materials to clinkering temperature.
(3) Decomposing the calcium carbonate.

(*b*) **Heat is recovered from—**

(4) The cooling of the hot clinker in the clinker cooler.
(5) The union of lime and clay at the clinkering temperature.

(*c*) **Heat is lost in—**

(6) Radiation from the kiln and cooler shells.
(7) The waste gases leaving the kiln exit end.
(8) Imperfectly cooled clinker leaving the cooler.

The operations referred to above may be considered in detail as follows :—The calculation of the heat required to

evaporate the moisture from the raw materials, and to superheat the steam formed to the temperature of the exit gases presents little difficulty.

It is not necessary to calculate directly the heat expended in raising the raw materials to clinkering temperature, and this indeed would be difficult, as the specific heats of the raw materials when approaching the clinkering temperature are not definitely known, and are also difficult to obtain. It suffices to remember that the heat expended in bringing the raw materials to clinkering point will be, under suitable conditions, restored in the clinker cooler. It is then only necessary to consider the losses due to defective cooling, such as imperfectly cooled clinker, cooler radiation, &c. The coal burned for the purpose of heating up the raw materials will only be that required to make good the losses such as those referred to.

The heat required to decompose the calcium carbonate is now generally accepted as equal to 774 British Thermal Units per pound.

The heat evolved by the union of lime and clay at the clinkering temperature is uncertain. Carpenter on Cement gives this as 29,868 B.T.Us. per barrel, or, say, 176,000 B.T.Us. per ton, which is equivalent to about 15 lbs. of coal per ton of clinker.

Methods are now available for estimating with considerable accuracy the radiation from kiln and cooler shells. The calculations depend on the surface temperature of the shell, which is measured by a suitable thermal junction.

The calculation of the heat lost in the waste gases presents little difficulty. A doubtful point is the specific heat of the gases at the higher temperature, such as 900° F., which is sometimes reached, and on this subject valuable experiments on specific heats of gases have been conducted by Le Chatelier and Mallard.

Heat Efficiency of Kiln.—From a consideration of what has gone before, it will be seen that the total heat or quantity of coal required to produce one ton of clinker in practice, is capable of fairly close calculation, and very close agreement can be made in this manner with the coal consumption observed in actual experiments extending over a long period.

Kiln Efficiency—Wet Process.—The efficiency of a rotary kiln is somewhat difficult to define in practice, but an estimate may be made in the following manner.

An ideal kiln may be assumed for reference purposes. This is assumed to work without radiation or cooler losses and without excess air. The water in the slurry is assumed to be 37 per cent., this being about the lowest value usually met with in practice. The temperature of the waste gases is taken at 212° F. Such a kiln working under the ideal conditions outlined above would have a total fuel consumption of approximately 16 per cent. reckoned on the clinker produced.

In practice, most kilns on the wet process will have a coal consumption nearly double, or 32 per cent. reckoned on the clinker. Hence, in the sense referred to above, the average efficiency of a modern process kiln (as compared with an arbitrary standard) may be said to be approximately 50 per cent.

Kiln Efficiency—Dry Process.—An ideal kiln using perfectly dry raw materials, working without radiation or cooler, or excess air losses, and having a waste gas temperature of 212° F., would operate with a coal consumption of approximately 8 per cent. reckoned on the clinker. In practice it is probable that dry process plants in this country working with a coal consumption of less than 20 per cent. are rare. Assuming this value the efficiency for a dry process kiln would be $\frac{8 \times 100}{20} = 40$ per cent.

On account of the increased temperature of the exit gases the efficiency of a dry process kiln when worked out on the lines referred to above is found to be less than that of a wet process kiln. The actual coal burned per clinker ton (which is the important point) is of course much less than on the wet process.

Utilisation of Waste Heat from Kilns.—The modern tendency is to use the wet process, with an increased length of kiln (and with a sufficient quantity of slurry lifters or similar appliances inside the kiln). The effect is to reduce the temperature of the waste gases below the point at which it would be economical to instal

waste heat boilers. In some of the earlier American plants, working with short kilns on the dry process, the temperature of the waste gases has been high enough, to render the use of such boilers practicable.

A considerable difficulty was the deposit of fine dust on the water tubes and elsewhere, but this appears to have been successfully overcome.

Whilst therefore waste heat boilers may be applied with success to some of the early plants working on the dry process, it is considered that both for the wet and the dry processes future progress is likely to be in the direction of reducing the temperature at which the gases leave the kiln. An equivalent reduction in the coal required for firing the kiln will naturally result.

The Rotary Kiln. Flue Gas Analysis.

CHAPTER XI.

From the coal analysis the value of the coal is arrived at, and from the flue gas analysis it is determined if combustion of the coal takes place in a satisfactory manner.

In an ordinary boiler furnace the air drawn through the fire by the chimney carries the supply of oxygen necessary for burning the fuel, air being a mixture of about 21 per cent. of oxygen and 79 parts of nitrogen by volume.

The nitrogen takes no active part in passing through the fire, and passes from the chimney in the same condition as it entered the furnace.

The oxygen combines with the carbon and hydrogen of the coal forming CO_2 and H_2O together with some CO if combustion is incomplete. These gases, together with the nitrogen and the excess air pass up the chimney. The percentages of CO_2, O_2 and CO present in the flue gases can be conveniently determined by analysis, and an examination of the result shows whether the fuel is being burned in a satisfactory manner.

The method used for the estimation of these gases is absorption in a special apparatus. A measured quantity of gas is passed into caustic potash solution to absorb the carbon dioxide (CO_2) then into alkaline pyrogallate solution to absorb the oxygen (O_2), and afterwards into cuprous chloride to absorb the carbon monoxide (CO). The apparatus generally employed for this test is the Orsat. This arrangement consists of a burette for measuring the gas, graduated from zero to 100 c.c. at the top, each c.c. being divided into 1-5th c.c. The burette is surrounded by a water jacket to protect the gases contained in it from external changes of temperature. The top of the burette is connected by capillary tubing with stopcocks to the three absorption tubes containing bundles of glass tubes, and the above-mentioned solutions.

A description of the **Orsat Apparatus** is as follows :—

(A) The level bottle connected to the burette by rubber tubing and two-thirds full of water.

(B) The graduated burette for measuring the gas.

(C) Rubber pump for drawing gases from the flue, this being connected to the three-way stopper D.

(D) Three-way stopper for connecting the flue to the pump of the burette.

(E) Absorption tube containing caustic potash.

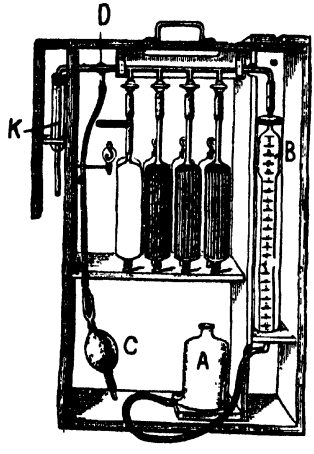
(F) Absorption tube containing alkaline pyrogallate.

(G) Absorption tube containing cuprous chloride.

(K) Tube for connecting *to* the flue.

Method of Working :—

Raise the level bottle A, and allow the burette B to fill



Orsat-Lunge's Apparatus for Furnace Gas Analysis.

with water to the *100* c.c. mark; then close the stopper D against the tube leading to B.

Now by means of pump C draw the gas from the flue through the tube K, till all the air in the tube has been

completely displaced by the flue-gas. Now open stopcock D to the burette B (making sure that the stopcock D is correctly placed to shut off C). Lower the level bottle, and allow the burette to fill with gas to the zero mark, then close D.

The burette now contains 100 c.c. of the flue gas to be tested. Now open the stopcock on E and at the same time raise the level bottle; this forces the gas into the caustic potash in tube E; lower the level bottle and the gas is drawn back to the burette. This is carried out two or three times, so that all the carbon dioxide may be absorbed. The last time the level bottle is lowered slowly so that the caustic potash rises to the height it originally stood at; the stopper of E is then closed and the level of the water in the burette is now read off, the reading giving the percentage of CO_2. The gas is now passed into F in the same manner to absorb the oxygen, and again into G, to absorb the CO (carbon monoxide).

Complete combustion of the coal is required to get the full heating value. From combustion the chief product is Carbon dioxide (CO_2), which should therefore show a good percentage in the flue gases. Carbon monoxide is formed by incomplete combustion of the coal, the presence of this gas therefore shows a wastage of fuel, as with additional oxygen the CO will burn to CO_2, thus producing more heat. The quantity of CO present in the flue gases should therefore be very small, and less than 1 per cent.

The formation of CO is often due to incomplete combustion caused by rapid firing.

The presence of Oxygen in the waste gases indicates excess of air. Air in excess of that required for combustion causes waste, because the cold air entering the boiler is heated up and escapes at a high temperature from the chimney top.

The best results are obtained when just enough air is supplied to burn the coal completely, thus preventing the formation of CO and consequent black smoke from the chimney top.

On account of the unequal distribution of the air passing through the fire it is generally found necessary to supply considerably more air than would be required if there was a more uniform mixing of the air with the coal in the fur-

nace. The table below, which is worked out for boiler coal of average quality, shows the relation between the excess air supplied to the boiler, and the percentages of CO_2 and O_2 present in the flue gases.

Excess Air per cent.	Air per lb. of Coal lbs.	CO_2 per cent.	O_2 per cent.	Remarks
0	10.4	18.3	—	The gas percentages are by volume
50	15.5	12.1	7.0	
100	20.7	9.0	10.6	
150	25.9	7.1	12.8	
200	31.1	5.9	14.0	

In the best boiler practice the excess air would probably be at least 50 per cent., corresponding to 12.1 per cent. CO_2 and 7.0 per cent. of oxygen in the waste gases. In less well arranged boilers the excess air may reach 150 per cent., the gas analysis then showing 7.1 per cent. of CO_2 and 12.8 per cent. of oxygen.

When very large quantities of excess air are indicated it is probable that much of the air does not pass through the furnace, but leaks into the flues between the furnace and the chimney. The gas analysis is usually taken near the chimney. The excess air can be conveniently deduced either from the CO or the O_2 present in the flue gases.

In the calculation of the excess air present when burning cement raw materials it must be observed that CO_2 is given off by the raw materials during the process of burning in the kiln, and this increases the proportion of CO_2 present in the flue gases.

It follows that in the case of rotary kiln exit gases a relatively high proportion of CO_2 indicates not only complete combustion and small excess air present, but also a type of kiln in which the heat of the coal is utilised in the most efficient manner. For the estimation of excess air alone in cement kiln practice, the percentage of oxygen present in the exit gases is a reliable indication.

For a rotary kiln on the wet process burning about 30 per cent. of dry coal, the gas analysis for various proportions of excess air will be approximately as shown in the table below.

Excess Air per cent.	Air per lb of Coal lbs.	CO_2 per cent.	O_2 per cent.	Remarks
0	10.0	26.7	—	The gas percentages are by volume
10	11.0	24.4	1.8	
25	12.5	21.7	3.9	
50	15.0	18.3	6.6	
75	17.5	15.8	8.5	

When burning powdered coal in a rotary kiln it is possible to cut down the excess air to 10 per cent.

Under these conditions and with a dry coal consumption of 30 per cent. the gas analysis, as shown by the table, would be approximately $CO_2 = 24.4$ per cent. and $O_2 = 1.8$.

The relation between the Oxygen present in the waste gases leaving the kiln and the excess air supplied will remain nearly constant for a wide variation in the percentage of coal consumption. The CO_2 shown by the gas analysis, however, is much more affected by changes in the coal consumption.

As previously explained, a correct regulation of the air supply in a rotary kiln will generally effect a saving where this matter has not previously received proper attention. The admission of too much air causes a loss of fuel owing to the heating of an unnecessarily large volume of air; on the other hand too little air leads to complete combustion of the coal. The latter is indicated by the appearance of black smoke at the chimney top.

With rotary kilns the danger usually to be feared is excess of air, as the general arrangements of the kiln tend in this direction. For estimating excess air the O_2 in the

waste gases is a much better guide than the CO_2, the latter figure being dependent in a cement kiln both on the excess air, and in the general efficiency of the type of kiln used.

Cement kilns differ in this respect from boilers. In the latter apparatus the proportions of CO_2 or O_2 in the flue gases are usually useful as showing the percentage of excess air present.

Correct air supply, complete recovery of the heat of the clinker leaving the kiln, low temperature of the exit gases and the recovery of potash are directions in which the cost of firing rotary kilns will be reduced in future.

The Rotary Kiln: Historical.

CHAPTER XII

Among the first Rotary Kilns for cement manufacture was one erected at Arlesey in 1887, and another at Hull, and soon afterwards a small kiln was erected at Penarth, and one at Grays. All these kilns were at first supplied with heat from gas producers. The Arlesey kiln, at that time perhaps the largest erected by Ransome—one of the original patentees—was 26 feet long and 5 feet in diameter. Subsequently the Stokes process was introduced in the industry, but none of these early Rotary Kilns, nor indeed any other Rotary Kiln, was at that time a practical or commercial success, the chief cause of failure being the absence of a suitable method of firing.

This has been since attained by investigators in the United States, and led to the adoption of an apparatus dealing with powdered coal with an air blast. Since that time various ideas in dimensions and designs have been incorporated in apparatus put upon the market by numerous makers of the kilns in many countries.

With the first introduction of the Rotary Kiln, however, it is doubtful if Crampton's patent of 1877 ever got beyond the experimental stage, and Ransome's ideas did not indeed get much further.

The following are some extracts from the first **patents** applied for in connection with the use of a Rotary Kiln for cement manufacture :—

T. R. Crampton, 13 Victoria Street, S.W., 22nd June, 1877, and 7th December, 1877.

(*a*) For the burning of Portland and other such-like cement in chambers or furnaces in which the cement

material is kept in motion either continuously or intermittently by mechanical agitation during the burning through them, substantially as described.

(*b*) The burning of Portland and other such-like cement in revolving furnaces heated by the gases resulting from the combustion of coal or carbonaceous material passing through them, substantially as described.

(*c*) The burning of Portland and other such-like cement in revolving furnaces heated by the combustion of air and powdered carbonaceous materia', substantially as described.

(*d*) The manufacture of Portland and other such-like cement direct from the raw materials being effected in a continuous operation.

F. Ransome, Lower Norwood, Surrey, 2nd May, 1885, for—

(*a*) The process of manufacturing Portland and other cement substantially as described, consisting in first reducing the cement material to a dry powder, according to the degree of fineness required in the burnt cement, and then burning such dry powder by keeping it in continuous movement whilst exposed to the heated products of combustion of a gas or other furnace so that the cement produced may be used without subsequent grinding.

(*b*) The process of manufacturing Portland and other cement substantially as described consisting in first reducing the cement material to a dry powder according to the degree of fineness required in the burnt cement, and then burning such powder in a slowly revolving chamber heated by the combustion of gas substantially as described.

Frederick Wilfrid Stokes, 13 Holland Street, Kensington, dated 10th March, 1898, for—

(*a*) The improved method or process of producing

cement by first forming the slurry into strips, sticks or sheets of regular or uniform transverse sectional area; then drying the same, and then subjecting them to intense heat in a revolving cylinder or furnace, while they are kept relatively in motion, substantially as and for the purpose, set forth.

(*b*) The described method or manner of simultaneously abstracting some of the heat from the burnt cement and heating the waste products of combustion or atmospheric air to be used for drying the slurry or for other purposes, as set forth.

(*c*) The employment of a conveyor lined with fire-brick or other refractory material and arranged to pass along a flue through which the waste products of combustion, or the wet slurry are caused to pass, substantially as, and for the purposes set forth.

(*d*) The consisting of a revolving cylinder lined with fire-brick or other refractory material (with or without the helical grooves) and so arranged that the waste products of combustion or atmospheric air will pass through the same, substantially as, and for the purposes, set forth.

(*e*) In my improved apparatus for burning cement, the provision for utilising compressed air for inducing currents of air into the regenerative chamber, substantially as, and for the purpose, above specified.

(*f*) The combination of a regenerative chamber, a revolving cylinder or furnace and a hood or casing for collecting the heat radiated from the said cylinder or furnace, substantially as described.

(*g*) The combination of an elevator, hopper and continuous feed with a revolving cylinder or furnace, substantially as, and for the purposes set forth.

(*h*) The improved method of forming the slurry into strips, or sheets, by forcing it in a plastic state through suitable apertures in an otherwise closed cylinder or

chamber by means of a piston, substantially as, and for the purpose, set forth.

E. H. Hurcy, Bethlehem, Pennsylvania, and **H. J. Seaman,** Catasauqua, Pennsylvania, 23rd December, 1898.—

(*a*) The process for automatically handling cement material, and continuously making and finishing Portland cement, substantially as specified and represented in the drawings.

(*b*) The process for continuously and automatically manufacturing Portland cement, which consists in preparing the cement material, roasting such material, cooling and pulverising the resultant clinker, and packing or storing the finished cement.

(*c*) The process for continuously and automatically manufacturing Portland cement, which consists in preparing the cement material, roasting such material, wetting, cooling, and pulverising the resultant clinker and packing or storing the finished cement.

(*d*) The described process of burning pulverised carbonaceous fuel in the presence of the cement material being roasted, which consists in injecting the pulverised fuel with a limited volume of high pressure air inducing a limited current of air at atmospheric pressure with the injected fuel and supplying the air necessary to support combustion of said fuel by a natural draft independent of the injected mixture.

(*e*) The described process of burning pulverised carbonaceous fuel in the presence of the cement material being roasted, which consists in injecting the pulverised fuel with a limited volume of high pressure air inducing a limited current of air at atmospheric pressure with the injected fuel and supplying the volume of air to support

combustion by a heated natural draft independent of the injected mixture.

(*f*) The described method of burning pulverised carbonaceous fuel in the presence of the cement material being roasted, which consists in injecting the fuel with a natural volume of high pressure air in the presence of a natural draft of air sufficient to support combustion of said fuel and allowing the air to carry the solid residue of combustion away without impinging against the cement material being roasted.

An interesting record, showing the possibilities of this kiln some thirty years ago, is contained in the description of the operation of the Rotary Kiln and the methods in use at the time, as reported by *The Engineer*, in the year 1887. This report of old standing, well descriptive of the manufacture of cement, by the then old and new methods, is here reproduced, and it will be seen that the article is peculiarly illustrative of what has actually occurred in the industry some thirty years after this report was written.

The following article has particular reference to the "RANSOME" **Rotary Kiln patented in 1885,** as then introduced in cement manufacture :—

The process of cement manufacture as it at present exists may be briefly described as follows :—Chalk is mixed with the requisite quantity of clay in a "wash mill," a large circular basin of brickwork, in which a series of heavy iron drags or harrows are caused to rotate by the motion of a central spindle, to which they are suspended by radiating arms. Water is run in, and the two materials gradually disintegrate and thoroughly mix. The mixture, in the state of thin slop, known as "slurry," runs out of the mill as it is formed, and is led off to large settling tanks or ponds, where it is allowed to remain at rest. The finely divided chalk and clay settle down to a stiff mass at the bottom, and the water is run off; a series of tanks

being so arranged that each one is ready for subsequent treatment about six weeks after it has been filled. The stiff mass, or "compo," left behind when the water has run off, is in many works removed and ground in an ordinary mortar mill, after removal from which it is loaded on to drying floors which are heated, either with waste heat from kilns or coke ovens or by special furnaces arranged for the purpose. A shed open at the sides protects the drying compo from rain, and it gradually solidifies into masses containing only a small percentage of moisture. These masses, varying from the size of a man's head to that of the fist, are placed in a kiln with alternate layers of coke. A fair-sized kiln will hold 70 tons of dried slurry, 15 tons of coke, and a small quantity of brushwood to act as kindling.

The fire is now lighted, and by the fourth day, or in, say, ninety-six hours, the 85 tons of material have reached a temperature closely approaching that of molten cast iron, and have also diminished in weight to about 30 tons, plus the weight of the ashes of the coke (about 15 cwt.). The kiln requires a day to cool, another day to unload, and the 30 tons of hard cement clinker is ready to be crushed, and then ground into a marketable product. In addition to the above process, there are two modifications of the preliminary part of it in use, known respectively as the "semi-dry" and "dry processes." These apply a less quantity of water in the mixing of the materials, the object being to do away with the settling tanks, and thus save space and labour. The treatment of the product in the kiln is, however, the same in all. It will be as well here to consider what has happened during the burning described above, and the first striking fact is the enormous decrease in weight. Firstly, the whole of the combustible portion of the coke has gone, mostly in the form of carbonic acid; secondly, the 70 tons of slurry has diminished to about 30 tons.

As far as the coke is concerned, assuming that 95 per cent. is carbon, the conversion of this into carbonic acid has involved the consumption of nearly 38 tons of oxygen, equal to nearly 166 tons of air; next the slurry has lost 40 tons, 38 tons being carbonic acid, and the atmosphere

has thus received from the kiln some 90 tons carbonic acid, 128 tons nitrogen, and about two tons of water in the shape of steam. When the last of the coke has burned away, there is left in the kiln 30 tons of cement clinker at a temperature of over 2,000 degrees F., the walls of the kiln have also, for some distance from the interior, been heated to the same degree, and before anything can be done this mass must have become cool enough for removal. It is obvious that the heat retained after the clinker is formed is lost during this cooling process. The value of this heat is considerable, and would probably be equal to nearly one-third of the total coke consumed. In other words, it may be assumed that to re-heat 30 tons clinker with the brickwork of the kiln would take five tons of coke. If this heat could be utilised it would effect a saving of, say, 2s. per ton of cement made. Another defect of the process arises from the large size of the lumps of slurry put in. Owing to this they invariably consist of (1) an over-burnt exterior skin, (2) a properly burnt inner part, (3) if at all large, of an under-burnt kernel. All these are of necessity ground together; it is impossible to do more than make a very rough selection for the mill.

The clinker now cooled and ready for the mill is extremely hard. It has first to be crushed by a Blake's, or other crusher to lumps about the size of a walnut, and these have to be ground under mill-stones. The extreme hardness of the material necessitates constant dressing of the mill-stones to such an extent that at least a quarter of the stones in a cement mill are always "up" undergoing this process. Thus, while the steam power required to deal with so refractory a material is a very heavy item, it is obvious that the maintenance of the mill in a thoroughly efficient state involves a constant and serious outlay. Another defect arises from the ash of the coke which being inextricably mixed with the cement clinker, is therefore ground with it, and acts so far as an adulterant.

Returning to the kiln, we find that each firing involves the heating of the cold walls of the kiln to the necessary temperature, and again cooling them to the point at which it can be unloaded and again loaded up. The kilns have to be built of enormously massive masonry, and withstand

the strains and racking caused by these constant violent changes of temperature. To keep them in repair involves very heavy outlay, and the first cost of a 30-ton kiln, of about 20 feet diameter, and 35 feet to 40 feet high, with walls tapering from about 3 feet, with its fire-brick lining, is extremely heavy. The ground space occupied by it is also very large.

It will be seen from the above that the processes of burning and grinding the cement are by far the most costly of all the operations involved in its manufacture, and that they are beset with defects, both scientific and practical, of a very serious nature. It is evident that if any great improvement is to be effected in the manufacture, to these portions of the process the most serious attention must be directed. It is therefore to this part of the work that Mr. Ransome has directed his attention, taking as his guiding principles economy of fuel, space and labour, and he has devised the following process :—

The slurry prepared by any one of the methods now in use is dried on a floor heated as usual or by waste gas from subsequent processes. The soft, friable, and easily crushed blocks are now reduced to coarse powder, and are then ready for burning. The old kiln is totally abolished, and in its place **a cylinder of boiler plate** is used. This is lined with good refractory fire-brick set in fire-clay and about every fourth row bricks are set up on end, thus producing a number of parallel longitudinal feathers or ridges extending completely through the cylinder from end to end. The outside of the cylinder is provided with two smooth rings or rails of iron. In the centre a third rail is wrought into teeth, into which a worm rotated at a slow speed gears. The two rails rest on friction rollers, and the whole cylinder being set at an angle with the horizon is caused to rotate slowly. This construction though sounding somewhat formidable, is in practice extremely simple, and similar machines, known as "black-ash revolvers," or "revolving black-ash furnaces," have long been, and are now, in daily use **in alkali works.** The cylinder is mounted on the top of a brickwork chamber divided by interior walls of brick. The two outer chambers are filled with bricks piled in loosely, chequerwise, so as to present a large surface.

We will now suppose a cylinder to be started, and describe the operations. A **gas producer** being in working order and delivering its gas at a regular rate is lighted, and the flame passes through the cylinder, which in the course of a few hours attains a white heat. The waste heat from the revolver has also passed through, and heated the right-hand division of the regenerator to a bright cherry red. A shunt valve is now opened, causing the waste gases to pass through the left-hand regenerator, while the gas from the producer is caused to flow through the heated right-hand chamber, and thus arrives at the mouth of the revolver already intensely heated. The result of this is that an immediate economy of fuel is produced, and to avoid over-heat it will be necessary to reduce the gas supply. During the whole operation the air necessary for combustion is also heated by passing down a separate division of the regenerator, where it receives heat from the walls of the outer compartments. As soon as the right-hand chamber begins to cool, the furnaceman reverses his shunt valve and the fresh gas is turned through the hot regenerator, while the waste combustion products are heating that which has cooled down. The effect of this method of working is thus to return into the furnace the heat which in ordinary methods of work goes up the chimney. No startling innovation occurs save in the application of the method to cement making. Regenerative furnaces are in use all over the world, and an intelligent furnaceman will learn how to manage one in a few hours.

We have now to turn our attention to the cement which, taken from the drying floor, we described as crushed to a coarse powder. The powder is lifted by any convenient mechanical arrangement to a hopper, placed at the upper end of the revolver; from this it falls in a steady shower through the flame, to the lower side of the cylinder, and lodges between the feathers. As the advancing side of the revolver rises it is lifted until the feather attains such an inclination as to shoot it off again through the flame to the bottom once more, but, owing to the incline, several inches nearer to the lower end. As the revolver moves on, this operation continues again and again, the powder is constantly lifted and shot through the flame in showers.

gradually getting nearer and nearer to the lower and hotter end of the cylinder, until at last it falls out into a receptacle at the lower end. In practice it is found desirable to rotate the cylinder at such a rate that any given particle of cement takes about thirty minutes to travel from one end to the other, during which time it has been lifted and shot through the flame about fifty times.

The powder has now arrived at the outside of the furnace, and having been delivered on to a floor to cool, is at once ready for grinding, that is, it is in the same state as the clinker after being seven days in the kiln. Unlike cement clinker, however, it does not consist of lumps weighing from 14 pounds downward, and as hard as granite, but a coarse, roughly agglutinated sand. Nor does it consist of an over-burnt skin, a properly burnt inner portion, and a possible under-burnt in most part, but if the operation has been properly carried out, each fragment has been heated to exactly the proper degree. How exactly this heat can be regulated is well known to all who have ever used a regenerative furnace. Again, the fuel used is gaseous, consequently no mixture of coke ash has taken place, and the cement is really and in fact what it professes to be.

So much would have been achieved had the new process been introduced with a furnace which had to be cooled in order to remove its contents, but—and here comes in a source of immense saving not only in fuel, but in repairs—this is not an intermittent but a continuous one. The revolver once started goes on night and day delivering its hourly quantity of properly burnt cement until its fire-brick lining requires renewing, an operation that has to be performed only occasionally. There is no constant loss of time and heat during cooling, loading and unloading, as with the kiln, but the hourly delivery of a ton of cement enables a works with two cylinders to turn out 336 tons of cement per week, a quantity that eleven kilns of the usual capacity could not produce in the same time. As a matter of prudent practice, the inventor advises that a spare cylinder with its regenerators should always be ready so that a works using two cylinders would, as a fact, have three. In such a works, as soon as a revolver was seen to

be in such a state as to require attention to its lining, gas would be turned on to the standing furnace, and in a few hours it could take up its duty, allowing the shut-down revolver to be repaired without any stoppage or diminution of the output. It is needless to say that the **re-lining of a kiln** is a very different matter, involving the loss of its services for many days.

The next question to be considered is the economy of fuel effected by the use of gas producers. Instead of consuming coke, these require only to be fed with slack, coal dust, or anything that will burn, fed in at intervals through a hopper. A two-cylinder works would require for its daily service probably one **gas producer**, capable of converting about 6 cwt. of slack per hour into gas. In addition, there should be one similar producer in reserve kept going at only one-fifth of its full power, its gas being utilised under the boiler or drying floors; it would thus always be ready in case of a breakdown to take up its full production and supply the revolvers in motion. These producers are chambers of brickwork, in which a portion of the fuel burning gasifies the rest, a small jet of steam being blown in to assist the operation. They consume the whole of the coal, nothing escaping but ashes, and thus alone effect a great saving in the stoppage of the waste of cinders inevitable under ordinary circumstances. Numerous first-rate makers exist, and the use of gas producers is daily extending in the country, even in places where coal is raised either on or in close proximity to the works. Their value as economisers is recognised in all furnace operations requiring intense local heats. In steel and glass-melting industries employing heats like that of the cement kiln, it is asserted that, coupled with the use of regenerators, a saving of 50 to 70 per cent. of the fuel formerly used is effected. The only use for which their value is disputed is for steam raising, though in works where they are required for other purposes, even here they would effect savings in fire-bars and in wages. Their cost is small, they occupy little room, they can be placed at any reasonable distance from the place where the gas is to be burnt, so as to be in close proximity to the coal siding, any labourer can shovel the slack into them, and they do not

require constant skilled supervision. As we have before stated, there are several forms in use, that of Wilson being one of the best.

On the occasion of an inspection of Mr. Ransome's experimental furnace at Grays, Essex, the revolver was furnished with gas from a small producer, built by the works bricklayer, which was gasifying about 2 cwt. slack per hour; this not only supplied the furnace, but the valve was partly shut down to control the gas, which was nevertheless in excess of what was required. In fact, the producer could have supplied two half-ton revolvers. There was accordingly here exhibited a consumption of about 2 cwt. slack per ton of cement produced, instead of the usual 7 to 10 cwt. coke per ton of cement clinker from the kiln.

The results derived from this plan of gas firing are therefore: (1) Possibility of working with regenerative furnaces, thus saving all heat passing from the revolver. (2) Use of about 3 cwt. cheap slack per ton of cement instead of 7 cwt. coke. (3) Complete combustion of all fuel, the steam injected being decomposed by the red-hot cinders, and producing carbonic oxide and hydrogen. In all ordinary furnaces great quantities of fuel are lost, and in the shape of cinders inextricably mixed with the ash or mineral matter of the coal. (4) The cement is kept entirely free from fuel ash.

In addition to these the revolver gives us the following advantages:—(1) Economy of space, two revolvers with their appurtenances, and one in reserve, covering 900 square feet, turning out the same weight of cement as eleven kilns covering 4,400 feet. (2) Continuous day and night working, and hence economy of fuel lost by necessary cooling, and subsequent reheating of the kiln walls. (3) economy of repairs, which are simple and cheap. (4) Less frequent need of repairs, as the continuous heat involves no racking like the alternate heating and cooling. (5) Economy in first cost. (6) Economy in grinding, a granular sand being produced instead of lumps of clinker, whereby crushers are quite abolished, and the wear and tear of the millstones greatly reduced. (7) Economy of hand labour. Revolver cement can be handled on the American elevator system. (8) Improved quality from (*a*)

non-mixture with fuel ash; (*b*) no over-burning nor under-burning. (9) Increased control over quality of cement, it being possible to stop, increase or diminish the flow of crushed slurry to vary its quality at any time. (10) Freedom from loss by accident. The ordinary kiln once charged and fired must burn out, whether charged wrongly or rightly, while, as before stated, any error in material can be rectified in a revolver as soon as discovered. (11) Perfect control of temperature. And lastly (12) power of varying temperature according to nature of material.

The results of the introduction of the Rotary Kiln anticipated in this early historical narrative have not, however, quite been realised even with modern plant.

Clinker Grinding.

CHAPTER XIII.

THE succeeding and final stage in the process of manufacture (after the calcining of the raw material) is the **grinding** into an extremely fine powder of the clinker which comes from the kilns, and this process has exercised quite a large proportion of ingenuity during the past few years. So much attention has of late been devoted to the manufacture, that, as regards the mixing of the raw materials in their due proportions and the proper calcination of the mixture so obtained, one brand of cement may be said to be very much like another, and provided the works are suitably designed and the various details of the manufacture carried out with care, it may be accepted as a general rule that cements differ only slightly in quality.

The question then arises:—what constitutes a high-class cement? To which question the answer is, that the true test of quality is not to be found in the brand, but in the tests which the cement can stand, and particularly the fineness to which the cement is ground, always provided, of course, that the materials are carefully selected and the various stages of manufacture are properly carried out. Engineers and architects giving any study to this question show a singular unanimity in declaring that a finely ground cement is far superior to one that is but coarsely or even moderately finely ground.

It is not intended, however, in this chapter to deal with the qualities of cement by grinding to various degrees of fineness, but merely to continue with a description of the processes generally employed. Indeed, even in this respect also it would not be possible to describe the entire number of mills in the numerous methods adopted at different factories, so a general description of the best



THE EDISON ROLLS FOR CLINKER GRINDING

known machinery at present used will doubtless suffice. It must be explained here, however, that in clinker grinding, not only is it necessary that a certain degree of fineness be attained to make a satisfactory quality cement, but also that as large a proportion of "**flour**" as possible shall be contained in the finished product.

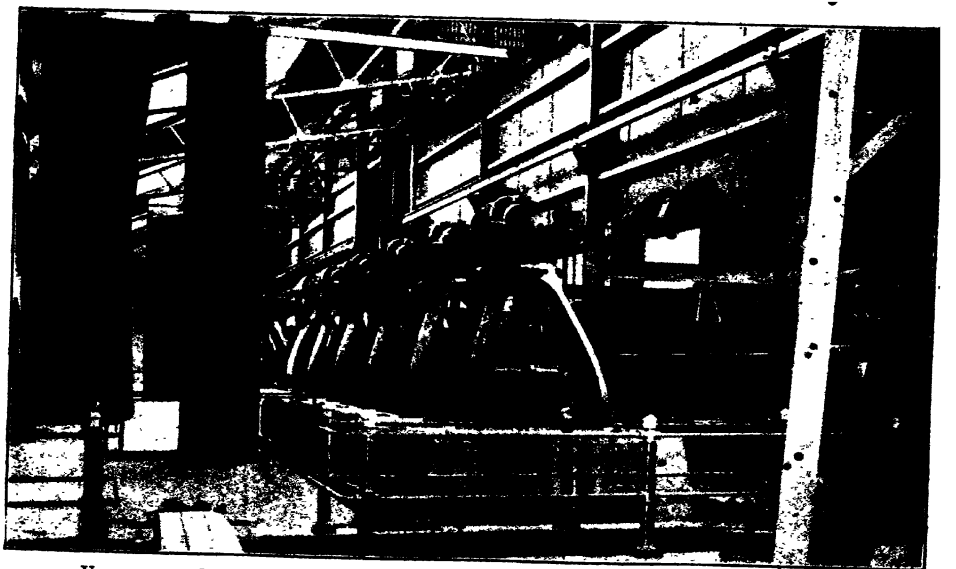
The first stage in the reduction of the clinker from the kilns is generally carried out by an ordinary **stone-breaker** or jaw crusher, which reduces the large lumps from the kilns to the size of about one inch cube, and from this crusher the clinker is conveyed to the fine grinding plant.

In the case of clinker from the rotary kiln the preliminary crushing is sometimes dispensed with, as in this case the clinker, when issuing from the kilns, is generally in the form of small rounded balls. In the event, however, of large lumps of clinker coming from the rotary kiln, crushing **rolls** are generally provided to do the same work as is carried out by the stone-crusher in the case of clinker from other kilns.

Until about twenty years ago the clinker grinding plant invariably consisted of **millstones,** but as the requirements of cement users became more stringent with regard to the fineness of the powder, so more economical and more efficient means have had to be provided, and these have resulted in the designing of new machinery for grinding the clinker into cement.

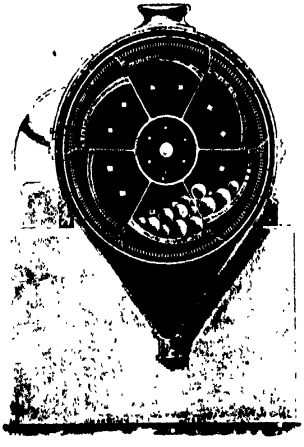
There is little doubt that the grinding of cement by millstones in ages past required a considerable amount of care and watchfulness, which, if neglected, added materially to the cost of manufacture, and it has been held that the cementitious quality of Portland cement ground by millstones was generally superior to the cement ground by other machinery, the stones being supposed to produce an angular rough grain; but upon examining the different samples of cement ground by various methods, it is observed that the difference, if any, in the shape of the grains is very slight indeed. The millstone for cement grinding is now, therefore, obsolete.

The question has often been raised as to whether or not certain systems of clinker reduction produced sufficient flour in the finished product, and in this respect it should



VIEW OF A CLINKER GRINDING PLANT, SHOWING BATTERY OF "GRIFFIN" MILLS.

be mentioned that some systems have methods of **crushing** and others of **grinding**, and there is a noticeable difference to the touch in the two products—the crushed product being less "velvety" than that from a tube or similar mill. This is accounted for in the operation of tube mills by a slow rubbing and rolling down of the cement, just as nature rolls and rubs down the fine sand found in sea and river beds. In the crushing method of reduction, however, the particles are broken into the most minute form, but even in its finished state the product is sharp,



VERTICAL SECTION OF BALL MILL.

clean and natural, the fracture exposing a nascent surface often chemically considered the best shape for reaction, but as to the practical results from the same cements ground by different processes to the same degrees of fineness the usual physical tests show, perhaps, one product to be as efficient as another.

In the better equipped and more modern works the preliminary grinding of cement clinker is carried out by the **ball mill** charged with heavy steel balls, and from this mill the coarsely ground material is conducted to a tube

mill containing flintstones, which finishes the fine grinding, previous to storing the cement.



The Tube Mill for Clinker Grinding

The ball mill consists of a short cylindrical drum, lined

with overlapping steel plates. The drum is mounted on and revolved by a steel shaft, which runs through it.

The drum contains steel balls from 5 inches to 2 inches in diameter, and the material fed to the mill is crushed and partly ground by impact.

When the material is reduced to a certain size it passes through slots or holes in the lining plates to a sieve surrounding the drum, which retains the coarser portion and returns it to the drum, and allows the finer portion to pass out of the mill.

For the finishing process—the fine grinding proper—the **tube mill** is employed, which grinds by means of the round flint pebbles or small balls contained within it.

The tube mill consists of a long wrought-iron revolving cylinder, carried on hollow trunnions at both the feed and delivery ends, and is about one-half full of rounded flint-stones. The coarsely-ground cement is fed from the ball mill into one end of the tube mill, and the rotary action of this mill, similarly to the ball mill, finely pulverises the cement as it passes through the falling flint-stones to the delivery end.

The finished cement is ground sufficiently fine to pass through a 76 by 76 mesh sieve with under 1 per cent. residue, or 10 per cent. residue on a 180 by 180 sieve, and although finer grinding is rarely demanded by the trade, it can be readily accomplished by the manufacturer (if necessary) at the expense of a reduced output from the mills.

In describing the tube mill, attention should be directed to the sketch of this mill.

Fig. 1 is a longitudinal section of a tube mill. Fig. 2 a plan of the mill, Fig. 3 a cross section of the drum, Fig. 4 a view of the feeding end, and Fig. 5 a view of the discharge end of the mill. The drum varies in size, the one illustrated having an interior diameter of 3 feet 11¼ inches, an interior length of 16 feet 5 inches, and revolving at the rate of 29 revolutions per minute. The drum consists of iron or steel plates half an inch thick, has cast steel ends, and is lined with hard cast-iron plates. The grinding balls or flint-stones are introduced through a manhole, whilst the material to be crushed is fed and discharged through the hollow trunnion cast together with the end walls. The

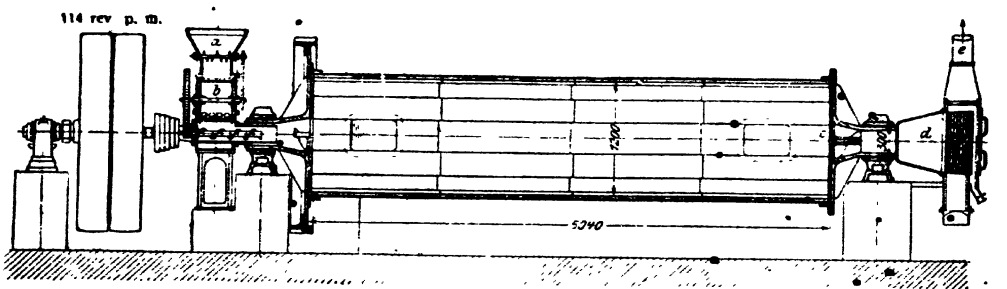


FIG. 1

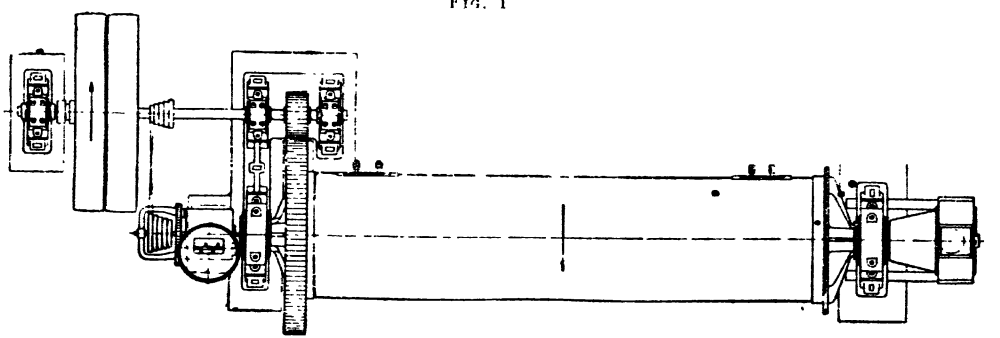


FIG. 2

material passes first of all into a hopper *a*, in which a studded shaft rotates. A longitudinally grooved drum *b* regulates the supply of material.

The drum is rotated by toothed wheel gearing and a pair of five-step pulleys at different speeds, and contains a few balls, which serve to shake the drum, and thereby ensure the emptying of its grooves. The material supplied is conveyed by a screw or worm into the hollow trunnion on the left side, Fig. 1, and then passes in the previously described manner into the drum, through the latter and through the grating *c* into the outlet trunnion on the right side. The inlet trunnion on the left side has blades which prevent the balls or stones from dropping out. The grating

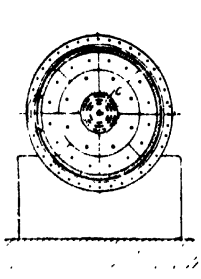


FIG. 3.

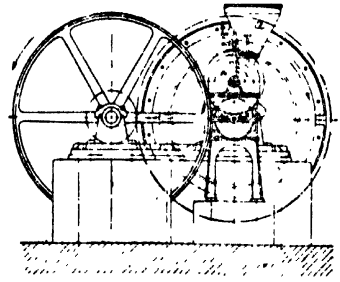


FIG. 4.

c is provided with slots of about one inch in diameter The material passes therefrom into a hopper *d* attached to the trunnion. A perforated screen is connected to the hopper. The slots of the screen are 5-16 inches in width and 13-16 inches in length; they permit the sufficiently pulverised material to drop through whilst very hard particles which are not crushed, and splinters of the flint-stone balls, are retained and subsequently drop out on the right side, Fig. 1. The sieve is enclosed by a casing, out of which the air is drawn by means of the pipe *e*, so that air enters all permeable parts, thereby preventing the exit of dust. A great advantage of the mills in which the

material is fed and discharged through the hollow trunnions of the drum, consists in the facility of rendering the same free from dust. If, however, the discharge of the material is effected through slots in the drum casing near

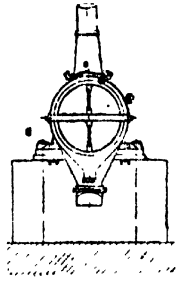


FIG. 5

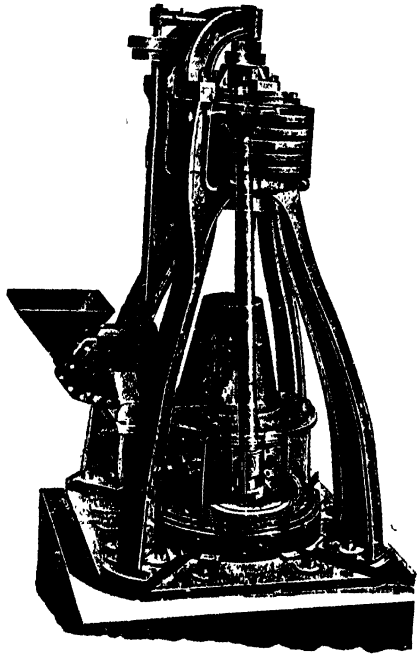
to the rear end, a comparatively tight closure is not possible, and the confinement of dust cannot be guaranteed by the suction of air.

The above described mill will produce the following results: —

Material	Preliminary Crushing corresponding to Sieve	Output per hour lbs.	Residue upon Sieves 180 / 180 %	Residue upon Sieves 80 / 80 %
Raw Limestone ..	50 / 50	7,150	20	0.75
Ditto, another kind ...	30 / 30	7,700	20	1
Ditto, another kind ..	50 / 50	7,900	15	1 1.5
Cement clinker ...	50 / 50	5,500	14	0.2
Ditto, another kind ...	30 / 30	5,500	20	0.6
Ditto, another kind ...	30 / 30	7,700	25	1.5

These tube mills are made with a diameter of 3 ft. 1⅔ in. to 6 ft. or more, and a length of 13 to 26 ft. Their

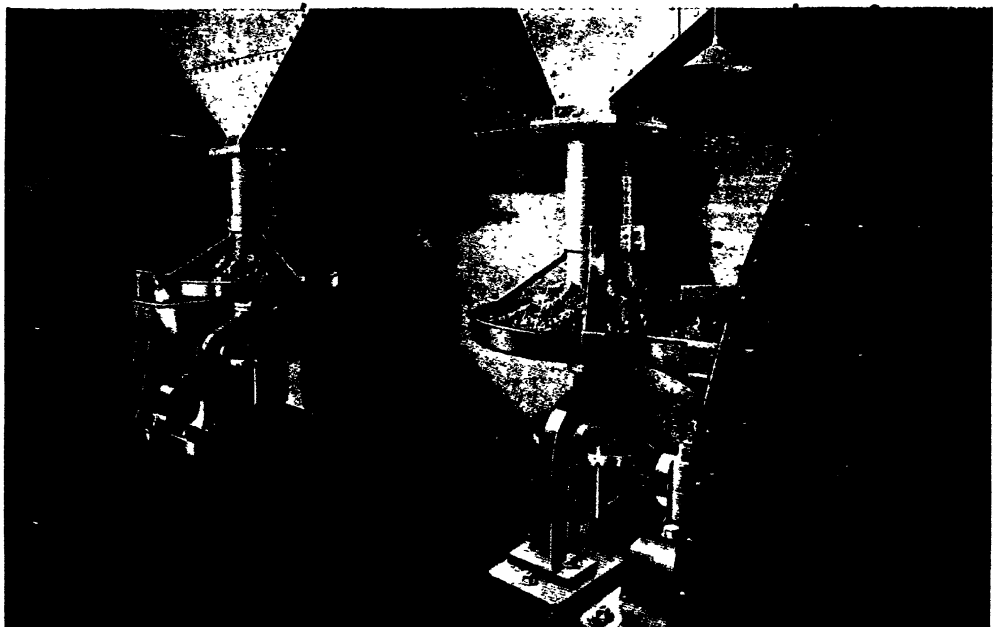
efficiency depends to a large extent on the kind of material treated and the degree of fineness desired and of course also on the initial size of grain, which is obtained by preliminary crushing.



"Griffin" Mill Arranged for Dry Pulverising.

Grinding mills also adaptable for use with clinker from the rotary kilns are the "Griffin" mills and Edison rolls —now often used in American works—and the "Kominor" mill, similar to the ball mill.

The Griffin mill takes the product as it comes direct from the rotary kiln, and the pulverising is done in one



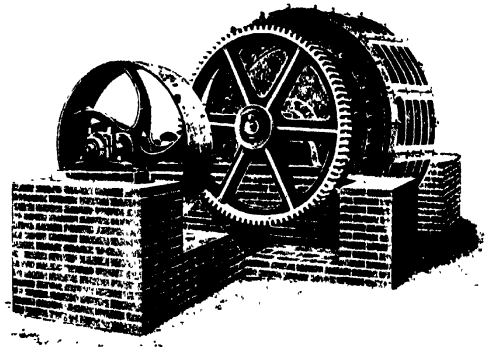
AUTOMATIC CLINKER FEEDING TO BALL MILLS

operation, and is discharged from the mill in the form of a finished product without the aid of any auxiliary sifting or separating device. This mill is an American invention that has also found much favour in modern factories in England. A heavy steel roller is attached to the bottom of a steel shaft which is provided at its upper end with a ball and socket joint. When the shaft is given a gyratory motion, the roller presses by centrifugal force against the inside surface of a heavy steel ring where the grinding takes place. The material which drops below the roller is thrown up again by steel blades that are also attached to the shaft, and when finally of sufficient fineness the powder escapes through sieves above the ring into a hopper below the mill. From here the finished product is automatically conveyed to the cement stores.

The **Edison** system of reducing cement clinker is that of a maximum of crushing with a minimum of grinding, and produces a very even product. The reduction is effected by rolls having corrugated crushing surfaces, which never touch in the act of crushing, a rather thick stream of material being passed through. These rolls run at a high peripheral speed, under great pressure, and are so arranged that the pressure can be changed when running, without altering the distance apart of the rolls, or the rolls may be set up to allow for wear of crushing surfaces, without altering the pressure. The crushing plates are of chilled cast-iron, and renewals are said to cost very little in the case of grinding cement produced from rotary kiln clinker.

The cement clinker in a size which will pass through a ¾-in. mesh screen is fed to the rolls, and after passing through them is carried in front of a series of blowers. Here the cement which is reduced to the requisite fineness is blown out and collected in settling chambers, whilst the coarser material is returned to meet the incoming clinker, and is again passed through the rolls. The capacity of this system is very great. A number of sets are now running each 30 inches in diameter by 8 inches width of crushing face, and producing from rotary kiln clinker 100 barrels (6 barrels to the ton) per hour of finished cement, of which 85 per cent. will pass through a 200 by 200 mesh screen, with a claimed consumption of power of about 10 h.p. hours per ton.

The illustration on page 161 is that of a self-contained set of rolls on this system, specially designed for a small output. These rolls will reduce cement clinker to such a fineness that 95 per cent. will pass through a 200 mesh screen with little increase in power.



"Kominor" Mill for Grinding Cement.

The **Kominor** mill is a development of the ball mill, as a coarse grinder, but both the Griffin and Edison mills combine the crushing action of the balls with the "flouring" action of the tube mill.

The fineness to which a cement is ground determines chiefly its usefulness for constructional purposes; for when a finer cement is mixed with the aggregate to be used in concrete it gives a higher average strength than that mixed with a coarser-ground cement. A fineness of about 90 per cent. passing a 180 by 180 mesh sieve is generally attained in modern practice. There is, of course, a point beyond which it is not economical to carry the fine pulverising of cement, since the same result can be obtained by using slightly richer mixtures on the work, but what this point is must be determined by experience.

The qualities of cement as regards tests of grinding are, however, dealt with in another chapter.

The Mode of Operation in clinker grinding by ball mills, and perhaps especially in tube mills, has been explained by recent investigations carried out by the plant makers and relative particularly to the tube mill. For a number of years past, experts have in vain endeavoured to explain precisely the effect of the operation of grinding by these machines, and this matter has been elucidated by the following description of the working and mechanical efforts of such plants :—

The operation of clinker grinding by ball and tube mills is carried out in a closed receptacle; the only visible evidence of the work accomplished being the incoming and outgoing material. As for the rest, one must resort to reflection, *i.e.*, to a comparison with known operations, which are deemed to be analogous to the operation in question.

Consequently, specialists have been of the opinion that in the case of the tube mill for instance the flints roll on the slope of the contents of the drum and shift in the bulk of the material to be crushed, thus crushing the latter between the flints and the drum, while the material passes from the feed to the discharge end of the drum, as the opening at the feed end is higher than at the discharge end.

Recent experiments have now rendered it possible to observe the operation in the interior of the drum. These observations give the result that the hitherto generally acknowledged opinion is fundamentally wrong, that especially the tube mill does not appreciably grind the material either on the slope or in the interior of the bulk, but crushes the material by impact or a so-called inclined beating action, and that a higher position of the feed opening as compared with the discharge opening is of no importance for conveying the material.

For the purpose of the experiments in question, a drum chiefly consisting of glass was used, and then a larger one was constructed in which the inspection of the interior of the drum was rendered possible by an interchangeable grating, and finally a drum of 39⅜ in. interior diameter, constructed similarly to the latter drum, was used for the experiments. The drum was first operated without

material and then with material of different kinds which would not give off dust, at varying speeds of rotation.

The discharge end of the drum, 39⅜ in. in diameter, was only closed by a wire grating, Figs. 1 and 2, so that the interior of the drum was clearly visible. The drum contained only rounded steel balls or flint-stones of about 2⅜ in. in diameter. The height of arch of this charge was about 17¾ in. when the drum was in a state of rest. The drum was then turned slowly by hand until a few balls or stones began to move on the surface of the bulk. Fig. 6 showing balls, represents this stage. Then the drum was set in operation.



FIG. 1.—39⅜ IN. DIAM. BEGINNING OF ROTATION.

At a rate of 21 and 23½ revolutions per minute, the balls rolled rather slowly down the slope and the height of the bulk increased a little. As the speed increased to 28, 30 and 32 revolutions per minute, the motion on the free side of the bulk became more lively and a more and more visible looseness took place; the height of the bulk increased to about 28⅝ in. Fig 2 is an instantaneous view of the operation when the drum was revolving at 32 revolutions per minute. The balls which are in contact with the drum below and on the ascending

side, are carried along without changing their position relatively to the wall of the drum, until they separate therefrom at a certain height and describe a somewhat distinct curve of projection. The balls or flints located farther inwards also do not change their position with regard to the drum when ascending, but a curve of projection is hardly perceptible on the descending side.

As the speed increased to 35 revolutions per minute, the contents of the drum were still more loosened, so that the height increased to 25⅝ in. The curve of projection of the balls which were raised when in contact with the drum, was clearly visible, and also the balls farther inward



FIG. 2.—39⅜ IN. DIAM. 32 REVOLUTIONS PER MINUTE.

separated in layers. On the ascending side, about 7⅞ in. from the wall of the drum, a few balls rolled in a hollow space of oval cross-section without intermingling with those moving farther outwards. The hollow spaces between the several layers on the descending side were rather clear and visible throughout, whilst on the ascending side the layers were arranged closely upon each other. Figs 3 and 4 are instantaneous views obtained with a drum 11 $\frac{13}{16}$ in. in diameter, and revolving at rates of 59 and 60 revolutions per minute. A comparison of both illustrations clearly shows how the looseness of the

contents of the drum increases with the speed. If the speed of rotation is still somewhat increased, the balls will form a solid ring revolving with the drum. A shifting of the balls is not perceptible. Fig. 5 is an instantaneous view of the interior of the drum, 39⅜ in. in diameter, when revolving at a speed of 55 revolutions per minute.

A large quantity of material producing no dust was now thrown into this drum. The material moved in the same manner as the balls, as was anticipated, then entered the hollow spaces between the balls and ascended and descended with the latter.

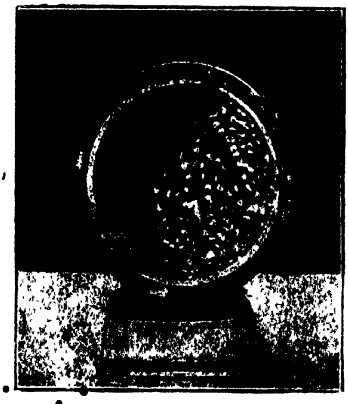


FIG. 3.—11⅝ IN. DIAM. 59 REVOLUTIONS PER MINUTE.

A difference was only noticeable in so far as the precipitated layers were less sharply demarcated and the particles of material were spurted laterally, especially at that spot which was hit by the mixture rushing down like a water-jet. Hardly any material was noticed in the contracted oval hollow space.

It has already been stated that a sliding or rolling motion between the balls, or between the balls and material, does not take place except at the striking point and in the oval-shaped hollow space. Since the latter is almost free from material, a crushing action—at least one of any import-

ance—can only take place at the striking point, where the falling balls are violently forced upon the material located between them and the balls which have been previously operating. The former substitute the shoes of stamps and the latter act as a substitute for the dies of a stamp battery. The crushing is effected by stamping or beating. The action, however, differs from that in stamp batteries in so far as the horizontal motion of the falling balls is opposite to that of the balls revolving with the drum.

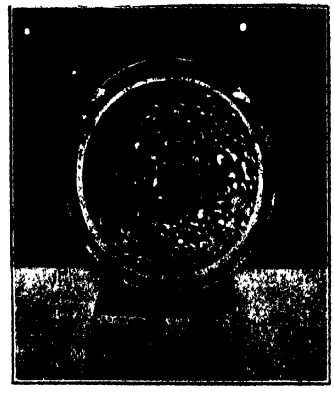


FIG. 4.—11½ IN. DIAM. 66 REVOLUTIONS PER MINUTE.

In Fig. 6 the curve of projection of the balls is shown as ascertained by calculation. It is assumed that the drum, 39⅜ in. in diameter, revolves at the rate of 34 revolutions per minute and derangements caused by atmospheric resistance, neighbouring balls and material are not taken into consideration. A ball situated on the wall of the drum is separated therefrom as soon as the component of gravitation which is directed towards the centre of the drum, *i.e.*, $mg \sin \psi$, has become equivalent to the centrifugal force $\frac{m v^2}{r}$: the ball then moves in a line of projection according to the equation :—

$$y = \frac{\cos \psi}{\sin \psi} \qquad x^2 = \frac{g}{2 v^2 \sin^2 \psi}$$

This is the line A, A_1, A_2. Fig. 6. The perpendicular velocity of the ball retards until it becomes zero in the vertex A_1 of the line of projection; consequently, the velocity in the direction of the path of projection will also be retarded, and the balls which were situated at some distance apart from each other on the drum casing, approach one another.

The balls located on the wall are represented in the figure at such a distance apart that they touch each other

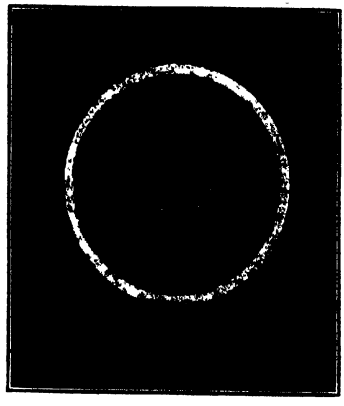


FIG. 5.—39⅜ IN. DIAM. 55 REVOLUTIONS PER MINUTE.

in the vertex A_1. It thus follows that the material between the balls is partly forced aside and that the balls, if arranged on the drum at a less distance apart from each other, would knock against one another and affect their speed. The shape of the path of projection would thereby be altered.

When a ball has reached the vertex A_1, without such derangement, it moves along the path of projection at an increasing speed, so that the distance between two neighbouring balls increases as shown in the figure, which corresponds to an accurate calculation.

A second layer of balls begins its path of projection at the point B, a third at the point C, &c. These paths of

projection are not so exact as A, A_1, A_2, because the balls, when moving with the drum are located in circles with less definite radii. As already stated, the current of material and balls following the path A, A_1, A_2, could, however, be very clearly distinguished from the current following the path E, B_1, B_2, during the experiments, so that

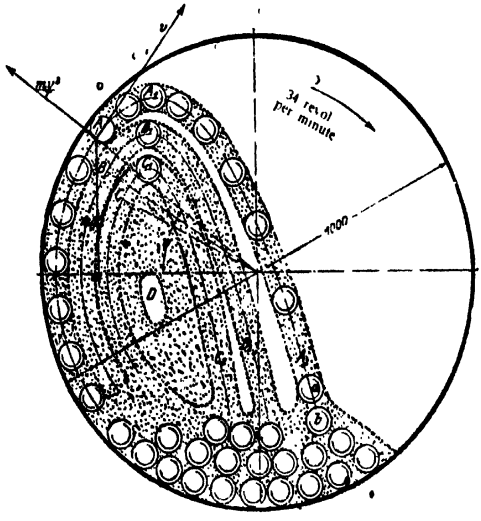


FIG. 6.—VIEW OF THE CURVE OF PROJECTION OF THE BALLS IN THE TUBE MILL.

it was possible to look between the same. The hollow spaces between other currents varied in size and clearness. The previously mentioned oval-shaped hollow space D changed its shape constantly as some balls were regularly moving therein.

There is no reason for the material to move differently to the balls, when it is not pushed aside by the latter.

Since balls and material, up to the starting points A, B, C, &c., of the paths of projection rest with regard to

the drum, a grinding action can only take place beyond these points. The decrease of speed between A and A_1, &c., however, is so small that up to the vertices A_1, B_1, &c., appreciable crushing cannot be expected. From the vertices to the striking points A_2, B_2, &c., the current lengthens, and crushing is impossible along these paths. Only when a downward rushing ball (*a*), Fig. 7 drops upon the material supported by a previously operating ball (*b*) or directly by the drum casing, the material will be smashed and, as the supporting surface

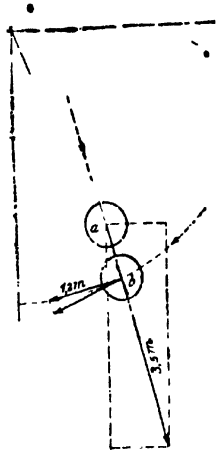


FIG. 7.

moves transversely to the direction of the projection, at the same time triturated.

Owing to the force of the strokes, the balls located near to the striking point will undergo a certain displacement, which, however, can hardly produce a crushing action of any importance.

The **crushing action thus depends** on the height of fall of the balls—*i.e.*, on the height of the vertex of

the path of projection over that point where the balls strike, on the speed of the drum, and on the weight and the number of balls. The speed of the drum must be so chosen that the paths of projection can be well developed. Weight of balls and height of their fall supplement each other in so far as they are factors of a product. Harder material requires heavier balls or a greater height of fall than softer material, and steel balls can have the same efficiency in smaller drums as flint-stones in larger drums. The larger the quantity of balls acting on a certain quantity of material, the better the crushing. If more

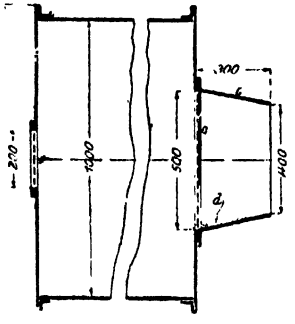


FIG. 8.

material of the same kind is to be crushed to the same degree of fineness within a certain period, the quantity of balls—and consequently the length of the drum—may be greater. The respective numerical values can only be obtained on the basis of extensive experiments.

In the further course of the experiments, the discharge end of the drum—Fig. 8 left side—was closed by means of a sheet-metal bottom (end wall), having in its centre a trellised opening of $7\frac{1}{8}$ in. in diameter. To the feed-end wall was fitted a hollow cone c of sheet-metal—Fig. 8, right side. The opening of this cone on the drum side—$19\frac{11}{16}$ in. in diameter—was trellised and partly covered by an immovable plate e secured to the machine frame, as

represented in Fig. 9. The material was now introduced through a manhole in order to quickly obtain the middle charge, and then the drum was set in operation at the rate of 32 revolutions per minute. A further quantity of material was thrown into the mill by hand-shovels. It was readily taken through the free opening of the grating, so that each shovelful rapidly vanished. On the outlet side, the material was discharged through the meshes of the grating, conformably to the curve of the projection. The discharge of the material thus took place at a considerably higher level than the feed opening.

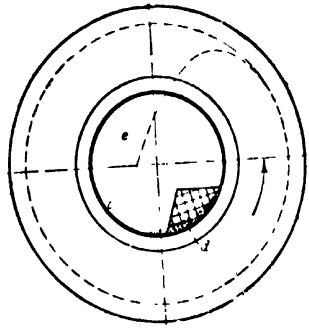


FIG. 9.

Consequently, the progressive motion of the material from the feed end to the discharge end—which is required by the tube mills—does not depend on a difference of height between inlet and outlet opening; there is no need to have the inlet opening placed higher than the outlet opening. It is only necessary that the inlet opening is provided at that place where the contents of the drum are loose enough to receive the material, or where the drum is empty, and that the moving contents of the drum pass by the outlet opening. The drum acts like an elevator, it lifts the balls and material innumerable times to a considerable height. In view of the sum of these risings, not

even the greatest possible difference in height between inlet and outlet end would be of any influence. An accumulation of the material cannot take place, because material and balls, inasmuch as they move with regard to the drum, form a very loose mixture, which readily receives the material.

Now by what means is the material compelled to pass through the drum? That material upon which a falling ball drops, will considerably spurt on all sides and be taken up by the neighbouring hollow spaces. If much material is located on the point of stroke, much material will be distributed therefrom, otherwise there is little effect. Consequently, those parts of the contents of the drum, which are richer in material, deliver more material than is returned by the parts poorer in material, whereby the proportions of the mixture are equalised. Owing to the great degree of vivacity at which the contents of the drum rise and fall—it was observed that some balls passed over their path twice during one revolution of the drum—this equalisation takes place very rapidly. At the discharge end, the material which spurts aside passes through the meshes of the latticed opening, whilst the balls are retained. Thus, on this side, the mixture will be poorer in grinding material, so that the said equalisation is directed hither.

In another trial, the feed-end wall with the conical attachment c, the grating and the screen e were removed and replaced by another end wall whose conical attachment was $13\frac{25}{32}$ in. in diameter, and provided with two helical blades. These blades revolve with the drum, and are so shaped that they prevent the balls from being thrown out of the mill, without impeding the discharge of the material. The phenomena did not undergo any appreciable change and no accumulation was noticed. The experiment, however, enabled a good view through the entire length of the drum, as the gaps between the helical blades permitted the light of an electric incandescent lamp to penetrate, and which sufficiently illuminated the interior of the drum. It was thus possible to look from the discharge end of the drum through the hollow spaces

between the falling layers and also partly beyond the falling material.

From these observations it appears that the grinding operation in a tube-mill is related to impact action which naturally depends upon the weight of individual grinding bodies and the path they travel; but from later experience it is also true that the surface action of grinding media must not be ignored in the mode of operation in clinker grinding.

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Motive Power and Factory Efficiency.

CHAPTER XIV.

HAVING now completed a detailed description of the various processes connected with the manufacture of cement and of the plant commonly used in the various departments enumerated, it is necessary to briefly describe the motive power for cement factories, and to consider finally the circumstances under which efficiency in production can best be secured in the co-ordination of the work of supervising factory running.

Motive Power.—It is unnecessary to dilate at length upon the power plants employed in cement factories, as this is a subject common to all industrial plants, and since there is no particular power generating plant especially applicable to cement factories, no attempt is made to urge one process of a subject of common knowledge in preference to others.

It might, however, be stated that a safe unit of motive power to adopt in cement works is two-horse power per ton of weekly output reckoned upon continuous night and day running and varying with the materials dealt with and the plant and process employed, viz. :—Weekly output × 2 = total I.H.P. required. This, as a rule would leave a margin for contingencies.

Whether an up-to-date steam generating plant with vertical or horizontal steam engines with direct or with electrical transmission be adopted, or gas plants with or without bye-product recovery, or whether plant of the turbo-alternator type is used, this is a department of the manufacture which is usually left to the discrimination, experience, and personal views of the cement maker in settling the layout of his own particular factory. Due regard must of course be paid to economy in installation, cost per h.p. hour, and more than either, perhaps, loss of output due to stoppage for adjustment and repairs with

plants that are very heavily worked, in night and day running, under conditions which are abnormal in regard to the dusty and gritty atmosphere arising from cement manufacture.

All of the above methods of power generation are adopted to-day in cement manufacture—except perhaps in the detail of bye-product recovery with gas plants—and each has its particular adherent and enthusiast, but since so much can be written upon a subject of this description and which, perhaps, after all is one of expediency and personal experience and opinion—applying similarly to all commercial undertakings requiring mechanical power—it is not proposed to enlarge upon this subject.

In regard to the broader issue of **factory control,** it should be added that the recording of efficiency in engines and machines has long received constant attention, but a deeper and still more important phase is the productive efficiency in manufacturing operations generally.

In any manufacturing industry the same principles of efficiency can be discerned as those pertaining to any engine or mechanism, but for this purpose the whole works must be conceived of as one machine, into which the raw material is entering at one end and from which the finished product is flowing at the other end. All the various machines, processes, and departments are but the wheels, links, and gearing of this one machine, and each should therefore be considered as a part thereof, and not as an independent unit, if high efficiency of the whole is to be attained.

In all classes of manufacture instances may be seen daily of one department, or even one machine cramping and reducing the output of the whole plant, and thus lowering its productive efficiency, in exactly the same manner as the efficiency of any engine or motor may be lowered through the bad design or workmanship of one part.

The problem of discovering and measuring the various obstructions, resistances, and other sources of loss which

lie hidden in all manufacturing processes is a most important one. In every industry there is a primary unit of measurement, which if carefully selected and applied, will prove of the greatest assistance in effective factory control. This unit for the measurement of works efficiency is the ideal output capacity of the dominating machine or department in that particular industry, together with the total capacity of the factory. By its employment the obstruction of the flow of work to and from the particular plant may be traced and measured.

The preparation of curves for the recording of daily outputs from each section of the factory will provide a weekly summary of results from which a monthly and annual diagram can be readily prepared, thereby showing at a glance a clear history of factory running and the causes of erratic output.

The ideal output capacity is adopted as the unit of measurement in order to obtain a measure which will not require altering from time to time, but will remain definitely fixed for long periods if not permanently. Its adoption does not connote the desirability or even the possibility of raising the works output to this standard of perfection, since, even if that were possible, the cost might exceed the gain. If the unit of measurement be called 100 per cent. the output of other departments (or machines) can be compared with it, when it may be found that sections such as quarrying, raw milling, burning and grinding, are individually giving results equal to 50, 60, 70 and 80 per cent. efficiency. The dominating department could easily produce, say, 90 per cent. if given the opportunity, but is throttled down by certain sections shown by the curve.

For instance, the summary curve may show that departments, say 1 and 2, are restricting and hampering the flow of work for all the rest of the plant, and that it is only by means of extra trouble and exertion that the work can be forced through these two at anything approaching the capacity of the rest of the plant. If suitable improvements may be made by which their capacities are raised to, say, 80 per cent., better, though not entirely satisfactory, results are obtained. Further attention to the weaker links may finally bring the whole plant up to a

uniform and harmonious rate of efficiency, but before the ideal 100 per cent. could be reached there would come a point beyond which it would not pay to go. This point can be decided only by careful attention, but is lower or higher according to skill in management.

In some cases special care is necessary in selecting the unit of measurement, so that it will satisfactorily cover and include all departments. The dominating department in cement factories, the requirements of which govern the output of all the other departments, and towards which the product of each of these flows, should be and probably is the kiln plant. Its output capacity, as it would be under perfect conditions of supply of the needful material, is therefore taken as the unit of measurement, and the actual average output of all the other departments is gauged by this standard. As soon as this is done it will probably be found that some department is specially culpable in holding back output.

The total output depends upon the running of a number of different sections of the plant, thus making it easy to see the cause of delay. The underlying principles hold good not only for a whole factory, but equally so for its departments. The ideal output capacity of the department at fault may thus be used as a measure of efficiency for the various machines which are working parts, and thus the deficiency may finally be traced to the faulty mechanism, design, or system of handling responsible. In fact, this is what is actually being done every day—more or less subconsciously perhaps—by every capable works manager.

It is not suggested that every department should be brought up to one inflexible standard of output, even were this possible. It is often wise to have considerable elasticity in the capacity of a department by means of efficient and economical storage to provide against breakdowns, but no plant should be kept normally running at a low output solely on account of its possessing one weak link, when the remedying of that one defect would both increase and cheapen the rate of output.

Output alone does not constitute efficiency, but it reduces costs, and where there are several different machines, processes, or departments dependent upon one

another, then the low output of any one of them will lower the productive efficiency of the plant as a whole no matter how excellent the individual efficiency of each separate item may be.

It is surprising how often resistance and obstruction to the flow of work are due to some comparatively trifling cause. A common source of hindrance is the placing of plant in positions relatively unsuitable, so that the material dealt with by them requires much unnecessary handling and also gets in the way of other efficiency. It is specially objectionable for work to be brought back against the outgoing stream, thus checking the flow of the freshly advancing material. So important is the relative position of one machine to another now realised to be that manufacturers must give special study to the planning out upon a board of these respective positions before agreeing upon a final lay-out.

The ideal system is, of course, such that the material goes on from machine to machine, or from process to process, without having to turn back upon or in any way obstruct that which is following. In some classes of work this course would follow a straight line while in others a zigzag path would better satisfy the requirements, each different process having its own peculiarities.

A frequent source of inefficiency is the running of any process intermittently instead of continuously. This factor is often neglected or not considered of much importance.

It is impossible to make suggestions for the infinite variety of plants in handling or conveying the material which occurs between machine and machine or process and process, but whether the material be heavy or light, hard or soft, wet or dry, the same principles hold good, and the same importance attaches to seeing that the mere handling is not setting up a back-pressure to the flow of work, and thus lowering the efficiency of the whole factory.

A daily or weekly curve record of defective working and of stoppages and breakdowns would prove of great help towards tracing the inefficiencies and weaknesses of any section of the plant. It would often be found that some little fault, defect, or stoppage, very trifling in itself, was being repeated over and over again from day to day or

even from hour to hour until it became multiplied into a figure of serious import.

An efficiency diagram might be plotted from day to day in each department, showing the benefits of improvements made and indicating the units still requiring attention. These diagrams could with advantage be prepared by works managers, where they would show at a glance the exact departments or processes most in need of attention in regard either to costs or to efficiency.

The curves are applicable not merely to new works specially planned and laid out in accordance with best practice. However old a plant may be the same principles apply equally well, and in many cases all that is necessary to attain good results may be a few alterations of relative positions of machines, conveyors or elevators and the provision of means for giving the plant free opportunity to work.

A plant with machines or departments of only moderate, but uniform, individual efficiency so arranged that a minimum of resistance is offered to the flow of work, is preferable to a plant with machinery of high efficiency but badly arranged. In the former case the whole plant can be worked smoothly at the highest efficiency its machinery will give, while leaving the management time and energy to raise the efficiency higher still; but in the latter case the high efficiency of the machinery is not allowed to display itself, but is hampered and wasted by the bad relative arrangement of departments or machines, while the energy and attention of the staff are consumed in overcoming the internal resistances which oppose the course of the products at every step.

Chemical Analysis.

CHAPTER XV.

THE **chemical analysis** of cements, if taken in conjunction with the mechanical and physical tests, affords much desirable information in the selection of a good quality material; indeed, it is almost necessary that both the chemical and mechanical examination of a cement should be made before the positive pronouncement of quality can be given, and when both have been ascertained, the suitability and efficiency of a cement for any given purpose can be at once accurately determined.

As to the progress which has been made in recent years in the testing of cements, it is clear that there has been a substantial improvement in the application of well-known processes, such as the mechanical and chemical modes of evaluation, but there has been little alteration in essence.

For example, there has been no successful attempt to appraise cement from its proximate composition, although that is the natural and right method of deciding its quality. The most pressing and practical of all the questions relating to the composition of cement is the determination of what is called "**free lime**," and many have been the prescriptions put forward for the purpose. On examination, all fail, and there is at the present time absolutely no method of determining by any chemical process whether a given cement contains free lime or not. There is no known method of extracting "free lime," if such exists, from cement without at the same time acting upon and decomposing the energetic unhydrated compounds which constitute the cement itself.

As to the value of chemical tests, it is not definitely known just what part is played by each of the compounds

that constitute present-day Portland cements, and chemical analyses do not explain the manner of the occurrence of these compounds. The definite aid which chemical analysis, therefore, may render at the present time in determining the quality of a cement is limited to ascertaining that the essential compounds are present in the proportions which experience has proved to be necessary or most desirable for the cement in question.

Neither complete nor partial chemical analysis of the constituents of hydraulic materials can therefore be ranked as infallible tests, or tests of primary importance. But chemical analysis may render real service in controlling the classification of a product concerning which there is reason to doubt the declaration of the manufacturer. Thus, a slag cement can be distinguished from a Portland by its composition, and also certain natural cements.

The actual chemical constituents of a well-made sample of Portland cement may be as under (example A), and the calculated composition of a cement burned from the sample of raw material referred to previously is also given (example B), viz. :—

(A)	Per cent.	(B)	Per cent.
Loss on ignition ...	1.22	... ...	
Silica (SiO_2)	21.44	... ...	25.28
Insoluble	.60	... ...	
Alumina (Al_2O_3) ...	6.75	... ...	6.23
Oxide of iron (Fe_2O_3) ...	3.17	... ...	2.99
Lime (CaO)	63.04	... ...	63.93
Magnesia (MgO) ...	.99	... ...	.71
Sulphuric anhydride ...	1.81	... ...	
Potash, Soda and loss	.98	Undetermined	.86
	100.00		100.00

There is a **change in composition** of the materials **during** the **burning** stage which is almost inevitably in the direction of reducing the lime factor, *i.e.*, making the cement more aluminous. This is caused by the impurities picked up during the calcining process, such as a slight mixture of kiln linings and the ash from the fuel used with

the kiln. These additions are chiefly composed of silica and alumina, and they affect and alter the theoretical composition of the cement as calculated from the raw materials used, by slightly increasing the percentages of silica, iron and alumina, and reducing the lime content.

The effect of **fuel ash** on the composition of cement as shown by analysis is illustrated by the following :—

The ash of the coke, or whatever fuel may have been used for the calcination of the raw material, being non-volatile, remains with the clinkered mass either wholly or in part, according to the type of kiln used. Part of it is fused on to the outside of the pieces of cement clinker and combines with it, and part may remain as a more or less insoluble powder or clinker, chiefly consisting of silicates of alumina and iron.

Such ash as is not carried away by the draught in the form of dust, being inseparable from the cement clinker, is ground up with it, and obviously must to a considerable extent affect the percentage composition.

In the types of fixed kilns usually adopted in England practically all the ash is thus left.

In the case of intermittent kilns, it takes about 8 cwts. of coke to calcine a ton of cement, and in the case of continuous kilns about 4½ cwts.

If we assume the coke to contain 10 per cent. of ash, this means that 0.8 cwts. of ash is left in the first case, and 0.45 cwt. in the other. This ash may have the following composition :—

Silica	51.00
Alumina and Iron Oxide ...	34 20
Lime	12 00
Undetermined	2 80
	100.00

and will affect the analysis of the average of the mass drawn from the kiln by reducing its percentage of lime.

If in the raw material the percentage of carbonate of lime be 76, and there be no volatile matter present other than the carbon dioxide belonging thereto, the amount of

lime in the calcined mass, if calcined by an ashless fuel, would be 63.94 per cent.

If calcined in an intermittent kiln under the conditions outlined already, the amount would become 61.86 per cent., and if calcined in a continuous kiln 62.77 per cent.

The British standard specification refers to the chemical composition of cement in demanding (1) that it shall contain no greater proportion of lime than is necessary to saturate the silica and alumina present; (2) that the percentage of the insoluble residue shall not exceed 1.5 per cent.; and (3) that magnesia and sulphur calculated as sulphuric anhydride must not exceed 3 and 2.75 per cent. respectively.

It also provides that the total loss on ignition (water and carbon dioxide) shall not exceed 3 per cent.

The percentages of **silica, alumina, oxide of iron** and **lime** contained in cements may vary to the extent of 1 or 2 per cent. from the figures given in the example A, and still constitute a good sound article, but magnesia and sulphuric anhydride should never greatly exceed the percentages given in the British standard specification.

Silica, lime and alumina must be considered the **essential elements** of the cement as they combine in the kiln at a clinkering temperature to form compounds having special properties in relation to the effect that water has upon them.

These compounds—silicate of calcium and aluminate of calcium—as they come from the kiln, and until they are wetted to form concrete, are perfectly anhydrous; that is, they are free from water, or, in other words, have no water chemically combined in their molecules. They are, however, capable of forming a chemical combination with water and in that condition are spoken of as hydrated silicate of calcium and hydrated aluminate of calcium.

The peculiarity of these compounds is that when in the anhydrous condition, they are more soluble in water than when in the hydrated condition, the effect being that when wetted with water to form concrete, the anhydrous substance is split up and a portion dissolves in the water; it then slowly combines with the water and the undissolved portion to form hydrated compounds, which, being insoluble, concrete the whole mass into a solid block. As the

solubility is but slight, this reaction takes place slowly, and it may be weeks or even months before it is complete.

This property is known as **hydraulicity,** and a hydraulic substance is one that will set under water or by the action of water.

The great aim of the cement maker is, therefore, to produce the maximum amount of these substances in his cement. For if on the one hand there is too much lime in the mixture a portion of it remains uncombined, and is a source of danger to the cement, as free lime expands and crumbles to dust on being hydrated, or " slaked " as it is commonly called, thus injuriously affecting the strength and stability of the concrete; so, on the other hand, if there is too much silica and alumina in the mixture, other compounds are formed which have no hydraulicity.

The proportion of **lime** in Portland cement may vary from 58 to 66 per cent. An analysis of Portland cement which shows a percentage of lime much outside the limits mentioned should, however, be submitted to very thorough tests before acceptance.

The **hydraulic index** of cement is found, as already stated, by the ratio of the silica and alumina to the lime present in the sample under investigation.

The **lime ratio** given by the British Standard Specification provides that the proportion of lime, after deduction of the proportion necessary to combine with the sulphuric anhydride present, to silica and alumina in cements shall not be greater or smaller (calculated in chemical equivalents) than is represented by the equation :—

$$\frac{CaO}{SiO_2 + Al_2O_3}$$

	Max.	Min.
	2.85	2.00

Molecular weight of Lime = 56
" " Silica = 60
" " Alumina = 102
" " Sulphuric anhydride = 80

An example of working this equation is as follows :—

Lime (CaO) 63.28% Silica (SiO_2) 21.6%
Alumina (Al_2O_3) 8.16% Sulphuric anhydride (SO_3) 2.00

Lime combining with 2.00 per cent. of sulphuric anhydride $= \frac{2.00 \times 56}{80}$

$= 1.40$ per cent.

$63.28 - 1.40 = 61.88$ per cent. Lime.

Thus—

$$\text{Lime (CaO)} = \frac{61.88}{56} = 1.10$$

$$\text{Silica } (SiO_2) = \frac{21.6}{60} = .36$$

$$\text{Alumina } (Al_2O_3) = \frac{8.16}{102} = .08$$

Then—

$$\frac{CaO}{SiO_2 + Al_2O_3} = \frac{1.10}{.36 + .08} = 2.50 \text{ Lime ratio.}$$

Up to a limit it may be said that the more the lime contained in a cement the greater will be its strength. This limit is reached, however, when more lime is present than will chemically combine with the silica and alumina, leaving some lime in the **uncombined** state—often termed "free" lime—occurring, generally speaking, when the percentage of lime is greater than the percentage of silica multiplied by 2.8 plus the percentage of alumina multiplied by 1.1.

Lime in slaking expands, so that an excess of lime over what will unite with the silica and alumina will cause the cement to expand or "**blow**," as it is often called.

If the percentage of lime is under the limit the cement contains silica-alumina in excess, tending to produce a contractive and weak cement. Unsoundness in cement is also caused by uncombined or loosely-combined lime resulting from coarse grinding or irregular mixing of the raw materials or under-burning of the clinker, but there is, however, as already stated, no method of determining the uncombined lime in cements.

Chemical analysis, therefore, if taken alone as the guide to quality, will seldom explain fully where unsoundness is concerned. Recourse must be had in this respect to the physical tests for soundness as mentioned in another chapter.

A Portland cement, containing high percentages of lime, requires high temperatures in the kiln whilst burning the raw materials. It is heavy and hard to grind and generally yields a **slow** setting cement. The rotary kiln has much improved the facilities for burning cement, and a general increase in the percentage of lime content in cements is now possible.

An increase in the quantity of **silica** in cements generally requires a proportionate increase in the lime content, with the result that the raw materials must be harder burnt, and the resultant cement will be of a high tensile strength. Silica is generally contained in cements to an extent of 18 to 25 per cent. It is one of the most useful constituents, but must not be in insoluble form.

Silica in Portland cement plays the part of an acid and combines under great heat with lime.

The function of the **oxide of iron** in cements, generally from 2 to 5 per cent., is to act as a flux in the kiln and cause the silica and alumina to combine with the lime at a lower temperature than they would without its intervention, and after that object has been effected the iron appears to play no further part. Its combinations are not hydraulic, and, consequently, have no concreting power.

The dark grey colour of cement is due to the presence of iron compounds, and when not in excess iron oxide does not affect a cement.

The amount of **alumina** in Portland cement is generally from 3 to 10 per cent., and its function is assumed to affect the initial setting of the material. A high alumina content, for instance, makes a quick setting cement, and a low percentage of alumina in cements generally defines their slow setting qualities. Alumina in Portland cement acts as an acid.

Magnesia is present in cements up to about 3 per cent., a limit which is considered to be harmless, indeed some experts consider that magnesia replaces lime in a cement,

and this theory is growing in favour, although other experts have maintained just the reverse.

The other constituents of cement, such as **sulphuric anhydride** and the alkalies **potash and soda**, are non-essential.

To ascertain the **insoluble residue** in a cement such as sand, clay, and inert material of any description, is useful in checking the existence of any foreign matter in the product, as a pure Portland cement should contain not more than 1.5 per cent. of such substances, which consist in such case chiefly of detritus from the kiln and mill linings and uncombined fuel ash.

There is no standard method for the **analysis** of Portland cement, and chemists vary considerably in their estimation of its constituents. This may be seen from the following results of the analysis of a sample of cement as carried out by the different methods of eighteen American experts working for a committee enquiring into the uniformity in the analyses of materials pertaining to the Portland cement industry.

	Silica	Alumina and Iron	Lime	Magnesia
Variation from ...	19.18	9.32	62.01	2.52
to ...	21.56	11.36	64.30	3.13

The **analysis** of Portland cement is generally conducted as follows :—

Half a gramme (500 milligrammes) of the finely powdered sample is carefully weighed in a platinum crucible and ignited, at first over a bunsen gas-burner and afterwards over a good blast lamp. It is then cooled in a dry atmosphere and weighed again. The loss of weight equals moisture and carbonic anhydride.

Silica (SiO_2) and Insoluble.—The same sample is now transferred to a large platinum dish and dissolved in hydrochloric acid and distilled water; about 5 cubic centimetres (5 c.c.) of the former and 10 c.c. of the latter. The solution is evaporated to dryness on a steam bath, moistened with dilute hydrochloric acid, and again eva-

porated to complete dryness. This operation may, with advantage, be repeated several times, to ensure that the silica, which separates from the acid solution of the cement, has become thoroughly dehydrated, and free from all trace of acid, otherwise a portion of it would redissolve on a further quantity of acid being added. The dry residue in the platinum dish is now digested on the steam bath with 10 c.c. of hydrochloric acid and 30 c.c. of distilled water until all is dissolved with the exception of a white powder, which consists of silica and insoluble matter. The contents of the dish are now further diluted with distilled water and filtered through an ashless filter paper, 9 centimetres in diameter, taking care to remove every particle of silica from the dish by washing it into the filter with boiling water. The filter paper which now contains the whole of the silica is thoroughly washed with a spray of hot water, the filtrate and washings being collected in a conical glass beaker.

The filter paper containing the silica is dried and ignited in a platinum crucible, at first gently over a bunsen gas burner until the paper is all consumed, and afterwards for five minutes over a blast lamp. It is then allowed to cool in a dry atmosphere, and weighed, the weight of the empty crucible being known; the weight of silica and insoluble is obtained by difference; the weight obtained in milligrammes ÷ 5 = percentage of silica and insoluble in the sample. The amount of insoluble ascertained, as further explained in this chapter, subtracted from this, gives the actual silica in combination.

Alumina and Oxide of Iron (Al_2O_3), (Fe_2O_3).—The filtrate and washings from the silica are heated to boiling in the glass beaker in which they are collected, about 5 c.c. of bromine water is added to ensure that the iron and manganese contained in the solution is fully oxidised, and the whole boiled until excess of bromine is dissipated. Solution of ammonia is now added in quantity sufficient to neutralise the hydrochloric acid present and until a distinct smell of ammonia is observed in the liquid after being stirred with a glass rod. The solution is then boiled gently until the smell of ammonia has nearly disappeared. A reddish brown, gelatinous precipitate will be observed in the

liquid. This consists of alumina and oxide of iron and whilst the liquid is still hot, must be filtered off on a filter paper similar to the one used for the silica. The whole of the liquid should be passed through the filter and the beaker in which the precipitation was made washed out with a spray of hot water (using distilled water) from a wash bottle. The filter paper and precipitate must then be washed, the filtrate and washings being collected in a large conical glass beaker, always taking care that none is lost.

The precipitate of alumina and oxide of iron is now re-dissolved in dilute hydrochloric acid (one part of acid to 10 of water) by passing about 10 c.c. of the dilute acid through the filter and collecting the filtrate in a separate beaker. Wash the filter with hot water, so that no trace of alumina and oxide of iron is lost, and make up the solution to about 50 c.c. Precipitate again by adding ammonia as before, and collect the precipitate on a fresh filter paper. Wash and add the filtrate and washings to those obtained from the first precipitation. Dry the precipitate and ignite in a platinum crucible over a bunsen gas burner, cool in a dry atmosphere and weigh; the weight in milligrammes ÷ 5 = percentage of alumina and oxide of iron in the sample. The object of the double precipitation is to free the precipitate from all traces of lime, which would otherwise contaminate the precipitate and cause a slight error in the analysis.

Separation of Iron and Alumina.—This operation may be carried out by either of the two following methods A or B.

(A) The precipitate of iron and alumina after weighing is fused with a little sodium carbonate (Na_2CO_3) and the fused mass is dissolved in hydrochloric acid (1-1 of water). To this solution add an excess of strong caustic potash (KOH) and boil for five minutes. Dilute the solution and filter off the iron precipitate, well washing with hot water, the filtrate being kept for the determination of the alumina.

The iron precipitate is dissolved in warm hydrochloric acid (dilute) and precipitated with ammonia, filtered, washed, ignited, and weighed as ferric oxide (Fe_2O_3).

The filtrate from the first precipitation is made acid with hydrochloric acid, and a slight excess of ammonia is added to precipitate the alumina. Boil off the excess of ammonia, filter, wash well and repeatedly with warm water. Ignite strongly and weigh as alumina (Al_2O_3).

(B) Instead of igniting and weighing the mixed precipitate of iron and alumina, this precipitate is washed back into a beaker and boiled with the strong caustic potash (KOH) solution, and above method carried out for the separation.

Lime.—The filtrate and washings from the last operation now contain the whole of the lime which is removed by precipitation with oxalate of ammonia. Add some ammonia to the liquid and boil. Whilst boiling, add about 25 c.c. of a saturated solution of ammonium oxalate, stirring during the addition and boil for one minute. The beaker is covered with paper and kept in a hot place (but not boiling) until the precipitate has settled down and left the liquid quite clear. It is then filtered and washed with hot water with the same precautions observed in the previous operations. The precipitate is burnt in the platinum crucible, at first very slowly over a low flame from the bunsen burner and afterwards at the highest temperature of the blast lamp, until the weight is constant and ceases to lose any more by further ignition; the weight in milligrammes ÷ 5 = percentage of lime in the sample.

Magnesia (MgO)—The filtrate and washings from the last operation, which will now amount to a considerable bulk, must be reduced by evaporation before the analysis can be proceeded with. For this purpose place the liquid in a large porcelain evaporating dish and boil gently until it is reduced to a bulk of about 40 c.c. If the liquid now is not quite clear, filter it, washing the evaporating dish and filter paper with a little water; cool the liquid by standing the beaker in cold water, add an excess of a saturated solution of phosphate of soda or microcosmic salt, and stir well with a glass rod, being careful to avoid rubbing the rod against the sides of the beaker. Now make the solution strongly ammoniacal by the addition of about 15 c.c. of the strongest liquid ammonia, cover the beaker with paper,

and stand it in a cool place for at least four hours or over night. During that time a crystalline precipitate of the double phosphate of magnesia and ammonia will have formed; filter and wash with a dilute solution of ammonia (10 per cent. of strong ammonia in distilled water), being careful to wash all crystals from the sides and bottom of the beaker; dry the filter and precipitate and ignite, at first very gently and afterwards at the highest temperature of the blast lamp. The precipitate will now consist of pyrophosphate of magnesia, and must be weighed in that state after the usual precautions of cooling in a dry atmosphere, after which the amount of magnesia contained in it can be arrived at by calculation. As 222 parts of pyrophosphate of magnesia contain 80 parts of magnesia, if the weight obtained be multiplied by the factor .36036, the product will equal the weight of magnesia contained in the 500 milligrammes of cement taken for analysis, and this number divided by 5 will give the percentage of magnesia.

Insoluble (Sand, Clay and Inert Material).—Two grammes of cement are weighed into a beaker, swirled with a little water to prevent lumps, about 100 c.c. 20 per cent. hydrochloric acid are added, and the beaker is gently warmed until all the cement is dissolved. Filter off the insoluble material, wash well, and place the filtrate and washings aside for the estimation of the sulphuric anhydride (SO_3). Now wash the insoluble back into the beaker from the filter paper and boil with a strong solution of sodium carbonate (Na_2CO_3), filter through the same filter paper, keeping the solution almost boiling, wash the insoluble back into the beaker and again boil with sodium carbonate. Filter, wash well with hot water. Ignite and weigh as insoluble.

This result subtracted from the combined weight of silica and insoluble first estimated, leaves the actual combined silica in the cement. Boiling with sodium carbonate dissolves out all the silica which may have been precipitated while dissolving the cement.

Sulphuric Anhydride (SO_3).—Bring the filtrate from the insoluble in hydrochloric acid to a boil, and pre-

cipitate the sulphuric anhydride by adding a 10 per cent. solution of barium chloride until no more white precipitate is produced. This white precipitate is sulphate of barium. The beaker has now to be kept at a gentle heat, but not boiling, for some time until the precipitate settles and the liquid above it is quite clear. It must now be filtered on an ashless filter paper and well washed with hot water; burn the filter paper and precipitate, when dry, in a platinum crucible over a powerful bunsen flame; cool and weigh with the usual precautions; 233 parts of sulphate of barium contain 80 parts of sulphuric anhydride, so the weight of the precipitate obtained in milligrammes must be multiplied by the factor .34335, and the product divided by 20 will give the percentage of sulphuric anhydride in the sample.

The analysis is now complete with the exception of alkalies, which are usually estimated by difference—that is, the difference between the sum of the various substances estimated above and 100 parts.

The results calculated to percentages are then tabulated as follows:—

	Per cent.
Loss on Ignition	
Silica (SiO_2)	
Insoluble	
Ferric Oxide (Fe_2O_3)	
Alumina (Al_2O_3)	
Lime (CaO)	
Magnesia (MgO)	
Sulphuric Anhydride (SO_3)	
Alkalies and Undetermined	
	100.00

The **dry atmosphere** referred to as necessary for the cooling of the crucible and contents after ignition, and before weighing, is best obtained by placing a jar or open wide-mouth bottle of strong sulphuric acid under a glass bell jar. The acid will absorb all moisture from the atmosphere contained in the jar. The crucible can be placed under the jar to cool near the sulphuric acid.

It is often desired by cement users to estimate the

amount of **lime only** in a sample of cement, and as this is a simple operation we may include the process under this heading.

Take half a gramme of the cement and dissolve it in an evaporating dish with a small quantity of concentrated hydrochloric acid; add a few drops of nitric acid and evaporate to dryness and bake well to ensure that all acid is driven off.

Redissolve the residue in dilute hydrochloric acid and filter off the silica.

To the filtrate add ammonia to precipitate the alumina and iron. Now add acetic acid to dissolve this precipitate of iron and alumina and boil the solution.

When boiling, precipitate the lime with ammonium oxalate in excess, filter, ignite and weigh as already described.

Mechanism of Setting and Hardening.

CHAPTER XVI.

THE mechanism of the setting of cement is still not precisely known, its complex constituents having as yet been scarcely isolated and their behaviour as individuals being almost entirely unknown. Until this detailed knowledge has been gained generalisation on such a subject must be confined to hypothesis.

The question as to what is the chemical cause and operation governing the process of setting is one of high scientific interest, and much complexity, and is one that really cannot be answered, but the theory is held that this is due either to a crystallisation or colloidal action, or both, and upon these points the following information is the latest that is available.

It appears to be accepted between the "crystalloid" and "colloid" schools that set cement derives its adhesive and cohesive qualities largely from the presence of low-limed compounds, or even of hydrous silica, although experts will not agree that the hydrolysis goes so far as to result in hydrous silica, as concurrently with the silica, hydrate of lime would be produced, and the two substances cannot long exist together, as fairly rapid combination takes place. Thomas Edison has explained to the author that he is a keen believer in the colloid theory, while Le Chatelier, the eminent French chemist, has always opposed the theory which attributes the hardening of cements to certain colloidal properties of the material. Other experts are equally as definite in opposition to these views. Le Chatelier has pointed out in a recent paper upon crystalloids against colloids in the theory of cement setting that in defining the word "colloid," if that be possible, Graham, the originator of the discovery of the bodies which are thus designated, had recognised the existence of two sorts of different solutions. The one kind gave on evaporation crystallised bodies called crystalloids

(sea-salt, sugar, &c.), the other gave amorphous bodies (gelatinous silica, gum, &c.). He further recognised that the crystalloids are diffused when dissolving and that the colloids are not.

More recently Zsigmondy showed with the aid of the ultramicroscope that the colloidal solutions were not true solutions, but merely suspensions of very finely divided, rigorously insoluble matter. These suspensions are opaque, milky or transparent, according to the fineness and the abundance of the particles so suspended in the liquid. When their diameter is inferior to the wave-length of light, they allow the light to wend its way through, somewhat as happens in the case of sound with screens of a limited size. The diameter of the true colloids approaches a millionth of a millimetre, and is always much less than a thousandth of one millimetre.

All very minute insoluble bodies give such colloidal suspensions; metals like platinum and silver when pulverised by the electric arc; finely crushed minerals like quartz, alumina, kaolin; completely insoluble chemical precipitates like the sulphides, notably nickel sulphide, or finally, the iron tannate used in the manufacture of ink.

But, is there cause to give a particular name to bodies, the only singular properties of which so far are insolubility and fineness?

These very minute bodies possess one quite special and very important property, however, which entitles them to being classed separately, that is the property of absorbing the soluble bodies which are held in solution by the liquid with which they are wetted. This absorption, which is frequent with chemical precipitates, is a very grave source of error in analyses. It is true that this property does not strictly characterise finely divided bodies; it manifests itself in identically the same way in porous bodies. Blood carbon, a true colloid, and wood charcoal behave in the same way. From this point of view, colloids and porous bodies must be classed together.

This absorptive power is a particular case of much more general properties which all very fine bodies possess. Chemically a body in the form of an impalpable powder behaves quite differently from the same body in the form

of big lumps. There is a special chemistry of colloids and porous bodies. These particular properties are under the immediate sway of surface tension.

The fact has long since been acknowledged that all chemical phenomena depend directly upon diverse forms of energy, heat, electricity, work. The chemical reactions of combustibles liberate heat; the reactions of batteries generate electricity; the reactions of explosives, work. Reciprocally the forces of energy—temperature, electromotive force, pressure—define equilibrium conditions and suppress or attenuate at the same time passive resistances which oppose the reactions, tending to bring the bodies back to the state of equilibrium. The whole chemistry is only a chapter of energetics.

There is, however, one form of energy of which chemistry does not speak as a rule, that is the surface energy which is measured by the product of the surface tension (or capillary tension) and of the free surface of the body. In magnitude surface tension is generally infinitely small when compared with the other forms of energy. Take the gram-molecule of water, 18 grams, and assume it to have the form of a sphere whose diameter will be 3.3 cm. In contact with its water vapour the sphere will have a surface tension of 0.08 grams per linear centimetre. The surface of the sphere being 34 sq. cm., its surface energy will be $0.08 \times 34 = 2.72$ gram-cm., or 2.72×10^{-5} kg. m., which corresponds to 6.3×10^{-8} kg. calorie. Now the vaporisation of a gram-molecule of water absorbs 10 calories, and its combination with lime liberates 10 calories. Its surface energy is therefore only equivalent to the one hundredth millionth part of that quantity of heat, that is to say, absolutely negligible.

If instead of taking the water in the form of a single sphere we divided it into a vast number of little globules, each of one-millionth of 1 mm., their surfaces and consequently their total superficial energy would become 330 million times greater, and thus equivalent to 2 calories; that is to say, the surface energy would be of the order of magnitude of the quantity of heat which water evolves in its various chemical reactions. The surface energy in that case would no longer be a negligible factor.

As regards solids in suspension in a liquid, there is no

lirect method for measuring the surface tension at the constant between the solid and liquid. Certain indirect considerations lead us to admit, however, that this tension may become a hundred times greater than that of water. The superficial energy will be increased in the same ratio and its influence will become appreciable for dimensions of the order of a ten-thousandth millimetre—dimensions much superior to those of chemical precipitates.

So far, Le Chatelier's considerations concern solely the grain size without paying attention to the greater or smaller solubility of the bodies in question. They do not yet furnish an immediate explanation of the usual insolubility of the colloids. A solid crystal ground down to very fine grains will certainly have all the properties of colloids as long as it retains its fineness; but it will rapidly lose the latter as soon as it is brought in contact with a liquid capable of partially dissolving the substance. The energy of dissolution will be increased by the whole superficial energy of the crystal; there will result an increase in the solubility of the fine crystals, and a supersaturated solution will be produced, from which larger crystals will soon be separated. This fact has been very clearly verified for the very fine precipitation of calcium sulphate and barium sulphate. In order to make these precipitates coarser, and thus prevent them from passing through the filters, such precipitates are kept hot for several hours before filtration.

Every soluble body, even if existing in very fine division, progressively loses its colloidal properties when it is maintained in contact with a liquid in which it is somewhat soluble; its transformation will be all the more rapid, the greater the solubility. It is only insoluble bodies, therefore, which can preserve the extreme state of subdivision which corresponds to the colloidal state. For the same reason, bodies which are properly called colloids never show any discernible crystalline form, because the crystalline orientation supposes a certain mobility of the material within the solvent, be it liquid or gaseous.

To come to the hydraulic mortars—Are the products of their hardening colloidal or are they not? Let us first take the case of plaster. When plaster has set, we can never discern any crystals in the material, even under the

strongest magnification of the microscope. That does not prove that no crystals are formed; perhaps they are too small to be recognised. In petrographic specimen plates less than 0.02 mm. in thickness, one can hardly distinguish crystals less than 0.005 mm. in diameter. By crushing the soft mass under the cover-glass we cannot get below 0.0005 mm. We are thus yet far away from colloidal dimensions. When the humid hydrated plaster is left to itself, crystals begin to become discernible at the end of several weeks; after several years they are sometimes visible to the naked eye. One can see them plainly, in the south of Algeria, on the roots of dead trees which have been transformed into gypsum. We can, moreover, make the crystals in hardening plaster visible from the beginning, when we gauge the plaster with alcoholic water (instead of pure water); the setting is then retarded and the crystals can better develop.

The calcium aluminates, which play an important part in the hardening of rapidly setting cements, are hydrated at the same rate as the plaster, and they give likewise a whitish mass in which crystals are not discernible. If, instead of gauging the aluminate with little water, we put a few isolated grains of the aluminate into a large volume of water, we observe that the grains soon become surrounded by long crystals of hydrated aluminate, which are always easily visible under the microscope and can sometimes directly be seen by the eye.

Calcium silicate alone causes a difficulty in these considerations, and this is important, because the silicate is the essential element in the hardening of Portland cement. The microscope has never enabled us to see hydrated calcium silicate. Its crystals are certainly very minute; numerous analogies prevent us, however, from denying their existence. The hydration is exactly the same as in the case of barium silicate.

$$SiO_2.3\,CaO + aq. = SiO_2.CaO,\ aq. + 2\,CaO,\ H_2O.$$
$$SiO_2.2\,BaO + aq. = SiO_2.BaO,\ 6\,H_2O + BaO,\ 9\,H_2O.$$

The crystals of hydrated lime are plainly visible, like those of hydrated baryta; they may attain dimensions of several mm. Crystals of barium silicate can always be seen with the aid of a lens, sometimes even with the naked

ye. By virtue of analogy we may say that hydrated calium silicate will certainly crystallise as well; but as it is nuch less soluble than the barium compounds, the crystals will be too minute to be discernible.

It is not impossible that, initially, the dimensions of these crystals are of colloidal order, as in the moment of formaion of the precipitates of barium sulphate and calcium oxalate. It has been shown, in fact, that this calcium silicate precipitate absorbs, in the moment of its formaion, a certain quantity of lime, which varies with the concentration of the liquid. In the course of time the limensions of the crystals must increase considerably, as n all similar instances. The mechanical resistance, on the other hand, does not diminish with time. Consequently, the colloidal state, which was possible at the beginning, has nothing, it is maintained, to do with the hardening of the cement.

In conclusion, the explanation of the hardening of cements which Le Chatelier gave thirty years ago does not yet appear to have been weakened, viz. :—

The anhydrous compounds of cements, not being in chemical equilibrium when in contact with water, are more soluble than the same hydrated compounds. For this reason they give rise to the formation of a supersaturated solution from which the hydrated compound may rapidly crystallise. The liquid, being no longer saturated can redissolve more of the anhydrous compounds, and the whole mass can thus crystallise progressively by passing through a stage of transitory dissolution in a limited volume of water, which, at any given moment, cannot maintain in solution more than a small portion of the total mass of salts present.

The crystals which are deposited from the su rsaturated solutions always appear in the shape of extremely elongated needles, which have the tendency to group themselves in spherolites about certain centres of crystallisation. The entanglement of all these crystals is the only cause of the solidity of the mass. They become welded with one another when the reciprocal orientation is suitable; they adhere by simple contact, as do all solid bodies when really resting upon one another.

Finally, friction at the points of contact, even in the

absence of all adhesion, suffices to produce a great strength of mechanical resistance, such as is found in all tissues and fabrics whose solidity has no other origin. The finer and the more elongated the crystals, the more numerous the points of contact, and the greater the frictional resistance. The greater the area of contact, the more developed the adhesion. The progressive growth of the crystal dimensions by the mechanism suggested increases the part played by adhesion while diminishing that due to friction. A material built up of very wide strips could be very solid if the strips were glued together at their contact surfaces.

While on this subject it should also be added that set cement contains hydrated lime, and no evidence has been presented to show that this hydrated lime is other than inert. We, therefore, have the anomalous position that, as the only known means of securing strength in their products, cement manufacturers are compelled to include the highest possible proportion of lime in their cements that is compatible with constancy of volume, while in the resultant concrete, a large proportion of the lime is nothing more than a diluent.

In their efforts to reach the maximum lime content, manufacturers are forced to grind their raw materials finely at considerable cost, and, the clinkering temperature of high-limed mixtures being high, the cost of calcination is also heavy. It is well then that the attention of chemists should be constantly directed to this anomaly, which requires the manufacture of cement to be burdened with an excess of a constituent costly to manipulate and which is ultimately found to be a diluent.

The production of low-limed cements has occupied the attention of many workers, but, unfortunately, the basis of their experiments and operations has invariably been blast furnace slag, with its inevitable content of sulphur as sulphide. So tainted at the source, such low-limed cements have seldom been judged upon their merits as cement, but have been condemned, and rightly so, on account of their sulphur content with its possibilities of disaster.

The elimination of sulphur from slag has not proved a

practical proposition up to the present, and it would tend to more rapid progress if this source of raw material for low-limed cement production were abandoned. The ordinary sources of raw material, such as chalk, limestone, clay and shale, could be used for low-limed cement production, and there would still be considerable scope for economy in cost of production, for three reasons: firstly, there would probably be no necessity for extreme fineness of grinding of the raw materials; secondly there would be less carbonate of lime to dissociate, and consequently less heat absorbed; and, thirdly, the low-limed mixture would require a considerably lower temperature of calcination.

The results obtained from slag cements offer much encouragement to the investigator aiming at the production of low-limed cement. These slag cements, containing about 45 per cent. of lime, sometimes have no difficulty in complying with the requirements of the British Standard Specification for tensile tests when mixed with sand, and for constancy of volume tests, and they thus satisfied two of the most important requirements of the cement user, but they are ruled out entirely by their content of sulphur, with their tendency to slow hardening, and the frequent faculty of losing their cementitious properties with age.

The sulphur difficulty can be avoided by abandoning slag and starting with "clean" raw materials, and the investigator of the future is then faced with problems of slow hardening and failure to keep, before low-limed cements can be made a marketable commodity.

It is, however, maintained by those who have devoted much time and labour to the subject that the major constituents of Portland cement are tricalcic silicate, dicalcic silicate and tricalcic aluminate. Of these constituents the compound tricalcic silicate is the one which hardens and develops the greatest strength within a reasonable time. This most important constituent, which is the one formed with the greatest difficulty, makes up only about 30 to 35 per cent. of an average normal Portland cement.

It may be said, therefore, that the essential process for the manufacture of Portland cement is the formation of this compound, and that any improvement in this process yielding an increased percentage of tricalcic silicate will

increase the cementing value of Portland cement. In order to determine the most economical process for producing tricalcic silicate in the highest percentages, it will be necessary to study the rate of formation of this compound in a series of mixtures of various substances; this, in turn, necessitates the determination of the equilibrium relations of tricalcic silicate of high temperatures in such mixtures. Such a procedure will lead sooner to the discovery of the optimum composition in various cases and for various purposes than the empirical trial-and-error methods that have hitherto prevailed.

PORTLAND CEMENT TEST SHEET

No.....192.....

SAMPLE of.....*brand.*

Received from.....

Tested at.....*by*.....

FINENESS :

Residue on 5,776 mesh.....%

32,400 „ .....% Flour.....%

Colour.

Setting Time tested by Standard Needle.

Initial.....mins.

Final ..... „

SOUNDNESS :

Expansion in Millimetres

"Le Chatelier" Test—Sample after 24 hours' aeration ... ...

„ „ after 7 days' aeration ... ...

TENSILE STRAIN :

Neat Briquettes gauged with.....% of water. State whether rammed or Plastic.....

	3 days	7 days	14 days	28 days	3 months	6 months
	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.
No. 1						
2						
3						
4						
5						
6						
7						
8						
Average						

Sand Briquettes, gauged 3 Standard Sand to 1 Cement with..... .....% of water, State whether Rammed or Wet.....

	3 days	7 days	14 days	28 days	3 months	6 months
	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.	lbs. per sq. in.
No. 1						
2						
3						
4						
Average						

ANALYSIS.

Silica (SiO_2)	
Insoluble Residue	
Alumina (Al_2O_3)	
Ferric Oxide (Fe_2O_3) ..	
Lime (CaO)	
Magnesia (MgO)	
Sulphuric Anhydride (SO_3) ...	
Carbonic Anhydride (CO_2) ...	
Water (H_2O)	
Potash (K_2O)	
Soda (Na_2O)	
Total ...	

REMARKS

If passes British Standard Specification.....

Sampling, Testing and Uses.

CHAPTER XVII.

In order to obtain the knowledge usually sought for as to the qualities of cements to be used, it is necessary that the samples which are taken for investigation should represent the whole quantity of the bulk.

Of all the materials of construction subjected to a system of testing, Portland cement is probably the most dependent on the judgment and skill of the tester, and with all the well-equipped laboratories it is still impossible to remove the most important factor of personal equation. In the **sampling** of consignments of cement and in making preparation for the testing and analysis of the same, the British Standard Specification does much to avoid the personal equation always evident in sampling bulk quantities, and it requires that :—

As soon as the cement has been bulked, either at the manufacturer's works or on the works in connection with which it is to be used, at the consumer's option, samples for testing shall be taken from each parcel. Each sample shall consist of approximately equal portions of cement from at least twelve different positions in the same heap when loose or from twelve different bags, barrels, or other packages, when the cement is not loose, so distributed as to ensure, as far as practicable, a fair average sample of the whole parcel, all to be mixed together and the sample for testing to be taken therefrom.

Before gauging the tests, the sample so obtained shall be spread out for a depth of 3 ins. for 24 hours in a temperature of from 58 to 64 degrees Fahrenheit.

In all cases where consignments are of 100 tons and upwards, samples for testing or analysis shall be selected as above from each consignment, either at the manufacturer's works or after delivery at the works where the cement is to be used. Payment for such tests and analysis shall be made by the consumer, the manufacturer supplying the cement required for the same free of charge.

The tests and analyses hereinafter referred to shall in no case relate to a larger quantity of cement than 250 tons sampled at one time.

In the case of sampling a large quantity of cement for analytical or other testing purposes, it is necessary that the small samples used in the analysis shall fairly represent the whole quantity. It is hardly probable, therefore, that a small sample, or even a large sample taken from one place in the manufacturer's store, or from one sack in the consignment, will have the average composition of the bulk, since at this particular point of sampling the quality of the cement might readily be of a better or inferior quality to the bulk.

It is well, therefore, to take small samples from various points, not merely upon the surface, where the cement may have become somewhat **air-set** by exposure to the atmosphere, but also in the centre of the heaps. This can be done by the use of a long **iron tube** which should have a sharp end and be fitted over a stick turned to fit the tube.

When thrust deep enough into the pile, the stick should be withdrawn from the tube, and then the latter should be pushed a little deeper into the pile of cement. The material then found in the end of the tube when withdrawn will furnish the required sample

The same method can be adopted in sampling numbers of barrels from one consignment. The samples of cement thus obtained should be mixed by turning over with a trowel after being spread out upon a table, and then the amount required for testing may be divided out.

The **American Society of Civil Engineers** in their standard specification stipulate " that the selection of the sample for testing is a detail that must be left to the discretion of the engineer; the number and the quantity to be taken from each package will depend largely on the importance of the work, the number of tests to be made, and the facilities for making them.

" The sample shall be a fair average of the contents of the package: and it is recommended that, where conditions permit, one barrel in every ten be sampled.

" All samples should be passed through a sieve having twenty meshes per linear inch, in order to break up lumps and remove foreign material; this is also a very effective

method of mixing them together in order to obtain an average. For determining the characteristics of a shipment of cement, the individual samples may be mixed and the average tested; where time will permit, however, it is recommended that they be tested separately.

"Cement in barrels should be sampled through a hole made in the centre of one of the staves, midway between the heads, or in the head, by means of an auger or a sampling iron similar to that used by sugar inspectors. If in bags, it should be taken from surface to centre."

In determining the good qualities of cement for constructional purposes, it is imperative that the product should possess at least two essentials—the qualities of strength and soundness; and the primary object of all **testing** is to determine whether the cement is satisfactory in these two important particulars. The other theoretical tests for fineness, specific gravity, time of setting and chemical analysis are of value only as the results give additional information as to the probable strength and durability of the material.

It is a rather remarkable fact, and one that makes the study of cement-testing most difficult, that the precision and accuracy possible in any one of the physical tests varies almost exactly inversely as the importance of that test. Thus, all would be willing to agree that soundness is the most essential quality for a cement to possess, and yet the ordinary tests for soundness are the most inexact, and until the Le Chatelier test was introduced, were the most dependent of all the routine tests upon personal judgment.

In the test for strength, which undoubtedly comes second in importance, we have all the variations due to the personal equation in mixing, moulding, handling and breaking, which make this test rather one of experience than one of exact treatment. In the time of setting, these inaccuracies are still further reduced on account of the simpler processes required. In the fineness test, which is far less important than any of the preceding, we reach the first test that is capable of some accuracy, or at least an accuracy that is measured more by apparatus than by experience in manipulation; and finally, in specific gravity, which, in itself, has almost no importance, we reach the

only test that can be considered as capable of precise determinations.

A few preliminary remarks concerning the other tests of cements may here be made, although dealt with at length in the following chapters.

Regarding the fineness of Portland cement, the coarser particles are practically valueless except as so much sand. The cement should at least **all** pass through a 50 × 50 sieve, and 98 per cent. through a 76 × 76 sieve. The finer the grinding the better the cement.

The **colour** of Portland cement should be greenish-grey. The specific gravity of Portland cement varies between 2.90 and 3.20.

When Portland cement is mixed with the necessary quantity of water, there is formed a plastic mass which hardens after a time. This transition is called "setting," and the time occupied is called "the time of setting." It is of great importance to know the setting time of the cement to be used. A greater quantity of mortar should never be mixed than can be used before setting begins.

It might here also be mentioned that in addition to testing cements by the chemical and physical tests according to well-known standards and theories, the average consumer generally tests the qualities of cement by **practical results,** preferring to watch the action of cement in work rather than rely upon the qualities as shown by the laboratory records. However, the "practical" test, be it said, is far more likely to give erratic results as to the qualities of cements, and with far more serious consequences than any of the recognised theoretical tests above mentioned, for there are innumerable ways in which a perfectly satisfactory quality cement, by faulty manipulation, may be made to appear otherwise, and there are again many aggregates which destroy the otherwise sound qualities of many products of proved first-class quality.

This practical test is often the parting of the ways between **manufacturer** and **consumer,** and in addition to being extremely unscientific, the test is fair to neither the one nor the other, and in the event of unsatisfactory concrete work ensuing, two distinct and definite courses are adopted by the cement maker and the purchaser—the one stating that he is only responsible for the quality of

his cement, and the other that he is not responsible for any faulty work carried out with the cement he purchases.

In view of what may be said as to the innumerable ways in which a good cement can be made unsatisfactory in use, the position which is thus brought about can generally be settled to the satisfaction of both parties by a little intelligent interest and labour in investigation, and the causes of faulty work or the failure of a cement in a practical test can far more often be brought home to **faulty use** than to faulty cement. The manufacturer often supports his position by having samples of the alleged faulty cement tested to the recognised standard of quality or specification, and there he considers his responsibility ends, which, perhaps, may be admitted to be true, but the cement maker, in addition, generally uses his further skill in explaining to a consumer where the " practical " test may have been faulty, merely for the benefit of his personal status.

It will have been found many times over that in numerous ways cement work may prove unsatisfactory from causes which are altogether beyond the control of the manufacturer, and have no relation whatever to the quality of the cement. Probably the most common cause of trouble is the failure to recognise the fact that the setting of Portland cement is a chemical process which may easily be retarded or destroyed by unfavourable conditions. Care, experience, and supervision are always necessary in the use of cement, and it is poor consolation for loss of time and money to throw the blame for faulty work on the manufacturer. He, may it be said, now works on much more scientific lines than has been the case in the past, and the cement maker is generally able to make a strong case for the defence where it may be necessary.

After a prolonged and careful enquiry in numerous quarters and over many years into the value of the practical test in the work as often carried out by the consumer in proving the quality of a cement, it has been found that in eighty-four per cent. of the cases specially investigated unsatisfactory concrete work has resulted through the faulty use of cements, and in addition a further eight per cent. of the failures were caused by the consumer ordering a special product which, either by its setting time or

tensile strength or some other special requirement which has been demanded, has not been suitable for the concrete or other work to be carried out with the cement.

There is much room for education on the part of the cement user. He needs to know, first of all, what he really wants in a cement—that is, what properties are of special value in his work. With this knowledge will come a better realisation of the necessity not only for uniform methods, but what is more to the point—uniform tests. When he realises that because one brand does not harden quite so rapidly as another, it is not necessarily inferior for the average piece of work; that the cement which fails to gain 12 or 15 per cent. over 900 lbs. from seven to twenty-eight days is not likely to suffer by comparison with average cements in any other way; and that when properly made and seasoned these cements are equally good for most ordinary work (although one or the other may be superior for some special work); when, we repeat, these facts are generally understood and appreciated there will be no reason for a manufacturer to modify his test methods so as to make a given showing—to obtain predetermined results—and uniform tests will be advanced a long way, a condition which the manufacturer will be the first to hail.

Some of the rudiments of the **use of cement** for concrete work are here referred to in passing:—

Mortar that has set should be thrown away, and on no account be retempered for use. It possesses no hardening properties, and it is therefore worthless.

The manufacturer has it in his power to make either quick setting or slow setting cement.

Slow setting cements are those that set after two or more hours. The slow setting cement is preferred to the quick setting cement for general work. Only for special purposes can a quick setting cement be used advantageously. In all cases the consumer should notify the manufacturer which he requires. By so doing a great many unjustifiable complaints will be avoided; but, above all, cements should not be ordered by consumers without the setting time asked for being that which is best calculated for and suited to the work in hand.

Portland cement is of all cements the least sensitive

to changes of temperature. Only during the process of setting can any injurious effect take place, and frost is then especially dangerous if the mortar is too wet.

In operations under water a quick setting cement is preferable. In plaster work, cornices, etc., and when working in cold weather, a cement should be used that is not too slow setting. In all other cases it is most advantageous to use a slow setting cement.

Portland cement as delivered to the consumer should be ready for immediate use, and should not, therefore, contain any **free lime**, since the lime (CaO) is changed on the addition of water to a hydrate of lime $Ca(HO)_2$, and the expansion which accompanies the change produces cracks in the hardened cement or concrete. This defect is only found in under-burned or carelessly manufactured cement, and all works of repute send out their product with the implied guarantee that free lime is not present. As a safeguard some engineers, when engaged on important work, subject the cement to a process of **aeration** during which the free lime present (if any) takes up moisture and carbon dioxide (CO_2) from the air and is converted into hydrate of lime and carbonate of lime ($CaCO_3$), whereby the danger of expansion in the setting cement from this cause is eliminated.

The following experiment illustrates the capacity of some cements to absorb carbon dioxide on aeration. Two samples of Portland cement were placed each in a wooden box lined with sheet zinc, stirred every day, and analysed periodically for carbon dioxide and loss on ignition. The following results were recorded :—

	Before Exposure	After 22 Weeks' Exposure
Sample A.		
Carbon dioxide ...	.40%	4.57%
Moisture and combined water	1.10%	2.61%
Sample B.		
Carbon dioxide ...	.48%	4.88%
Moisture, etc. ...	.72%	2.34%

Under different climatic conditions the same cements gave very much lower results. The graph showing the rate of absorption of carbon dioxide was a straight line.

Portland cement, therefore, readily absorbs moisture from the atmosphere, and soon becomes lumpy or even a solid mass if exposed to damp. It must, therefore, be stored in a dry place.

Pure Portland cement may be used as **mortar** where the work remains permanently under water or in wet ground or in enclosed spaces. Otherwise, Portland cement must be mixed with a greater or less quantity of sand.

The sand must be sharp, clean and graded from coarse to fine, the coarse preponderating.

In making mortar the water should be clean and free from acid, alkali or organic impurities.

The quantity of sand mixed with the cement is determined by the character of the work to be undertaken. One or two parts (by volume) of sand to one part (by volume) of cement is only employed when extraordinary hardness, great durability and water-tightness are desired. Three parts of sand to one part of cement is the proportion used for ordinary masonry.

The amount of water used in mixing the mortar is to be increased or diminished according to the requirements of the cement. General practice requires cement to be mixed with as little water as possible, and the mortar to be brought to the proper consistency by a prolonged and vigorous mixing.

Mortar is made by first spreading out the measured quantity of sand; then the necessary amount of cement is spread over it, and the mass stirred together until a uniform mixture is obtained; finally, the correct quantity of water is added, a little at a time, through a rose, and the mortar thoroughly worked.

For **concrete**, the selection of the aggregate is of importance, because upon its strength and hardness depend the strength and hardness of the finished product. Where a good concrete is required, the stone employed should not be inferior in point of hardness to the cement mortar used, therefore it is preferable to use granite, gravel or hard limestone. The use of sandstone, brickbats and generally

any porous stone should be avoided if the concrete is to be reinforced or is likely to be exposed to the influence of frost.

The size of the coarse material should be varied according to the purpose for which the concrete is to be used, but should not exceed about 2½ inches in diameter; it must, however, in all cases be graded, in order that with the addition of the sand and cement the voids may be properly filled and the maximum density secured.

In order that the concrete may afford the necessary strength with the greatest economy the proportions will vary according to the materials used and the purpose for which the concrete is to be employed. Where a very good concrete is required, as, for example, in ships, important reinforced work or water tanks, the proportions adopted may be one part (by volume) of Portland cement and three parts graded aggregate. For ordinary purposes, a mixture of one part Portland cement, two parts sand, and four parts coarse material is often employed; whereas, for heavy walls, foundations and other structures, where actual strength is not so important as weight and stability, the proportions may be 1 : 3 : 6.

In order to produce a dense strong concrete, every void must be filled and every particle of sand and stone coated with cement. If this end is to be accomplished, the mixing, both in the dry state and after the addition of water, must be very thorough, as it is obviously highly important. In large jobs this is usually done by machinery, but where it is to be done by hand, the sand and cement should first be thoroughly mixed. The stone should then be added, and the whole again turned over two or three times. Finally, the calculated quantity of water must be added gradually through a rose, and the whole mass mixed again very thoroughly, and *used immediately*, since concrete begins to set within twenty or thirty minutes.

No definite rule can be given as to the amount of water to be used, as this will differ according to the nature of the aggregate, the purpose for which the concrete is to be employed, and the temperature at the time of mixing. The necessary quantity must be determined by experiment; it will be such, however, that when the concrete is placed and tamped a little water will just flush to the surface. It

should be noted that in reinforced concrete it is essential that the mixture should be sufficiently plastic to pass between the reinforcement and to surround thoroughly every portion of the steel, though not so wet as to allow any dripping of the material.

Great importance is attached to the careful tamping of concrete until water appears upon the surface, as it is only by this means that maximum density and hardness are secured.

Where concrete or any form of cement work has to be laid on a foundation, the latter should be thoroughly wetted before the former is placed.

The qualifications of a good **aggregate** are that it must be clean, graded, hard, and, where occasion demands it, fire-resisting. Clean—*i.e.*, free from clay, loam or vegetable matter, in order to secure the perfect adhesion of the cement; graded, for the reasons already given; and hard, to impart strength and to resist abrasion. In buildings, or wherever the material is liable to great heat, all aggregate that expands or splinters under the agency of heat, such as limestone and dolomite, should either be avoided altogether or, if used, broken into small pieces.

The aggregates most suitable for making good concrete are, as already stated, granite, gravel and crushed stone; but other materials, such as coke-breeze, clinker and broken brick, may be used for certain purposes with satisfactory results, *provided care be taken that they contain no deleterious matter.*

Coke-breeze is often used because of its cheapness. It is a good fire-resisting material, but the chief objection to it is that it is porous, and may allow moisture to penetrate the concrete, which renders it unsuitable for use where a damp-proof structure is required, and would be fatal if the concrete contained any embedded steel work. Where, however, impermeability is not essential, as, for example, in partition walls, or in the inner leaves of cavity walls, coke-breeze may be usefully employed, provided it is clean, free from sulphur and from pieces of unburnt material.

Clinker, like coke-breeze, is light and porous, and produces a concrete which is not so strong as that made with

heavier and denser coarse materjal. It may, however, be used for certain purposes provided it is well washed and free from deleterious matter.

Broken brick, although porous, is often quite a good material, and produces a fire-resisting concrete, but great care should be exercised in choosing it, as some bricks contain sulphur and unslaked lime. Only a hard-burned variety should be selected, and any plaster remaining on the bricks should be removed and excessive dust avoided.

Slag must be used with great care, as it contains more or less sulphur which causes the corrosion and expansion of the reinforcement. It may be freed from sulphur by burning, after which it should be well washed, and allowed to weather for a considerable period in the open air.

Some **clays,** if well burnt are, like brick, suitable for concrete where porosity is not a disadvantage, and burnt gault is said to have great fire-resisting qualities.

Ground chalk may be used as an aggregate where great strength is not required and where impermeability is not an essential.

The ways in which cement work may be spoiled by the user are numerous, and it may be useful here to summarise the most common mistakes: --

(*a*) The use of too much water during mixing, or water carelessly applied, or an insufficient quantity of water.

(*b*) The use of dirty aggregate—*i.e.*, containing earthy matter, clay or loam.

(*c*) The use of water containing impurities.

(*d*) The use of an insufficiently graded aggregate—*i.e.*, one containing only coarse material or one with too much sand.

(*e*) The incomplete incorporation of the aggregate with the cement through insufficient mixing.

(*f*) The use of regauged concrete which has begun to set.

(*g*) The rendering of cement work on dry foundations and without thoroughly saturating the surface with water.

(*h*) The repeated "floating" or trowelling of cement surfaces.

Up to recent years, it was generally understood that concrete work could not be carried out during **frosty weather,** and, therefore, upon the approach of frost all operations of this nature were suspended. In modern practice, however, such means are adopted as enable concreting to be carried on throughout the year.

Heat hastens the hardening of concrete; cold delays it. Experience has shown that freezing has no injurious effect upon concrete that has first had an opportunity of hardening under favourable conditions for at least 48 hours. If, before early hardening has taken place, concrete is allowed to freeze and thaw at short intervals, its ultimate strength will be impaired; but, as a rule, it will not show any serious effects from having once been frozen if, after it thaws out, it is not again frozen until early hardening is complete. It is far better, however, to protect the newly laid concrete from frost for two to four or five days (according to the degree of cold) than to expose it to the possibility of freezing.

Where concrete work has to be carried on during frosty weather, some method of **heating the materials** previous to mixing is essential.

The **sand and coarse aggregate** may be heated in various ways. One method, suitable for small jobs, is by the use of an improvised stove, such as an old iron boiler or iron cylinder. A fire is lighted within and the materials piled around and over it, so that the "stove" is in the heart of the pile. The materials should be frequently raked over, so that all parts of the pile may be equally heated.

Another method is that of piling the aggregate on coils of perforated steam pipes, and supplying a steady supply of steam directly to the materials. If the pile is at the same time covered with a tarpaulin or other suitable protection the heat is prevented from escaping.

A third method is to heat the materials in the concrete mixer by means of steam jets.

Heating the **water** presents no difficulty. The simplest method is to carry out this operation in vessels over a fire, but on large jobs the mixing water is usually heated by live or exhaust steam from the boiler; where considerable quantities of water are required, live steam is to be preferred.

The **cement**, forming but a small proportion of the mixture, need not be heated.

The materials should be heated to such a degree that when the concrete is placed the temperature will be about 50-60°. A temperature of about 100° F. will generally prove sufficient; if higher, there is danger of injury to the aggregate.

After the concrete is placed in position it should be carefully protected.

The operation of heating the materials has a double advantage: the concrete is largely safeguarded against frost, and the hardening process is accelerated.

A method sometimes adopted for the prevention of freezing, and which had a considerable vogue when this work was first attempted in cold weather, is the addition of salt to the mixing water. The principle underlying this method is that certain substances, such as common salt and calcium chloride, when added to water, lower its freezing point. In the case of salt, each unit per cent. added lowers the freezing point one degree.

This method cannot be recommended. In the first place, the addition of salt delays the hardening of the concrete, whereas it is most desirable that this process should be hastened; then there is danger of the corrosion of the reinforcing steel in the presence of salt; and finally, when the cold is extreme such a high percentage of salt would be necessary that the final strength of the concrete would be seriously affected.

The production of a **waterproof** concrete is the aim of cement users in a large part of their work, and that this aim may be accomplished is unquestionable.

The natural method of making a waterproof concrete is to select an impermeable aggregate, so to arrange the sizes of its particles and the proportion of cement that, after thorough mixing, no voids exist. Concrete so made, with

conscientious workmanship and reasonable precautions, will be waterproof without the addition of any compound whatever.

While no authoritative statement has been issued in this country by a responsible technical body on the subject of waterproofing concrete, the American Bureau of Standards after carrying out tests with forty different compounds has published the following conclusions :—

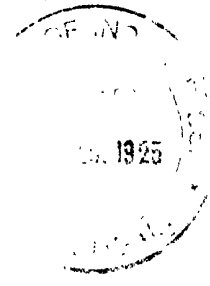
"Portland cement mortar and concrete may be made practically watertight or impermeable . . . to any hydrostatic head up to 40 ft. without the use of any of the so-called 'integral' waterproofing materials, but in order to obtain such impermeable mortar or concrete, considerable care should be exercised in selecting good materials as aggregate, and proportioning them in such a manner as to obtain a dense mixture.

"The addition of so-called 'integral' waterproofing compounds will not compensate for lean mixtures, nor for poor materials, nor for poor workmanship in the fabrication of concrete. If the same care is taken in making the concrete impermeable without the addition of waterproofing materials as is ordinarily taken when waterproofing materials are added, an impermeable concrete can be obtained."

With the same proviso, concrete is perfectly suitable for the storage of certain **mineral oils.** The coarse material and sand used must not be of a porous character, and must contain no foreign matter whatever; they must be well graded from coarse to fine, so that there is no excessive proportion of particles of any one size; and the coarse material and sand must be used in such proportion one to the other as will give when mixed with the cement the least bulk of concrete for given volumes of each of the materials when measured separately. Just sufficient water must be used for mixing the concrete to ensure a thoroughly plastic mass, which will flow into place, and which can be consolidated with light tamping.

While it is now well established that concrete may be used for the storage of mineral oils, its suitability in the case of vegetable and animal oils is not so clear, since these

are liable to contain fatty acids, which attack the concrete. As the result of many tests, it may be stated that while many vegetable oils may be safely stored in concrete receptacles, the material is not suitable for the storage of animal oils unless the surface is treated with some acid-resisting product.



Fineness and Sieving.

CHAPTER XVIII.

POSSIBLY the first enquiry the cement user makes as to the quality of the material under notice takes the form of a test for fineness; thus in passing through the manufacturer's stores, or when the first bag is opened on the contractor's works, the cement is mechanically and half-unconsciously passed through the fingers in order to ascertain the fineness. The coarse particles in a cement can thus be readily perceived, and the practised tester is, in this way, able to gauge approximately the residue present in the sample.

The test of Portland cement for fineness of grinding is somewhat indirect, for assuming that a finely-ground cement is sounder and stronger in test for tensile strength with sand briquettes, then such qualities are unmistakably determined in those tests. And, further, the apparatus used and methods adopted in the fineness tests are not considered satisfactory; but since strictly precise results are not absolutely essential, the present methods are perhaps sufficiently accurate for the purpose.

In many of the tests for fineness which have been made from time to time it appears that the fine grinding of cement very often reduces the tensile strength in neat briquettes, but increases the strength when a mixture of sand and cement is tested.

For the past few years there has been a growing desire on the part of engineers for a much finer cement, and it may be said that the principle of fine grinding is now in the ascendancy—an improvement in the manufacture greatly in favour of the consumer both as regards economy in use and reliability in result. Much has been written

concerning the value of fine cements, but it is necessary to explain further what results are to be expected in the testing and by the proper use of a finely-ground material; for, unless carefully dealt with and thoroughly understood, a fine cement is calculated rather to prejudice the cement user against what is undoubtedly a superior article.

Years ago the usual fineness called for in a cement was about 10 per cent. residue on a 50 by 50 sieve (*i.e.*, 2,500 meshes to the square inch), and much valuable work has been done with a cement of this fineness in the past, but to-day the fineness is more likely to be :–

Sieve	50 by 50	...	...	Residue Nil
,,	76 by 76	...	...	,, trace.
,,	100 by 100	...	...	,, 3 per cent.
,,	180 by 180	...	...	,, 10 per cent.

The British Standard Specification calls for a cement that shall be ground to comply with the following conditions of fineness :—

"One hundred grammes (4 oz. approximately) of cement shall be continuously sifted for a period of 15 minutes on each of the undermentioned sieves and in the order of succession given below, with the following results :—

" The residue on a sieve 180 by 180 = 32,400 meshes per square inch, shall not exceed 14 per cent.

" The residue on a sieve 76 by 76 = 5,776 meshes per square inch, shall not exceed 1 per cent.

" Air-set lumps in the samples may be broken down with the fingers, but nothing shall be rubbed on the sieve.

" The sieves shall be prepared from wire cloth, and the diameter of the wire for the 32,400 mesh shall be .0018 inch, and for the 5,776 mesh .0044 inch. The wire cloth shall be woven (not twilled), the cloth being carefully mounted on the frames without distortion."

Too much attention cannot be paid to fineness of grinding, for the finer the cement the more intimately can it be incorporated into the aggregate used for the concrete, and therefore the stronger will be the ultimate work. At the

same time, however, the use of a fine cement necessitates a more careful study of its properties.

As has been stated, the test for fineness derives importance from the fact, apparently well established, that, other qualities being equal, fine cement has a much greater sand or aggregate carrying capacity than coarse cement has; that is, it will show an increased strength with the same charge of sand, or equal strength with a greater charge.

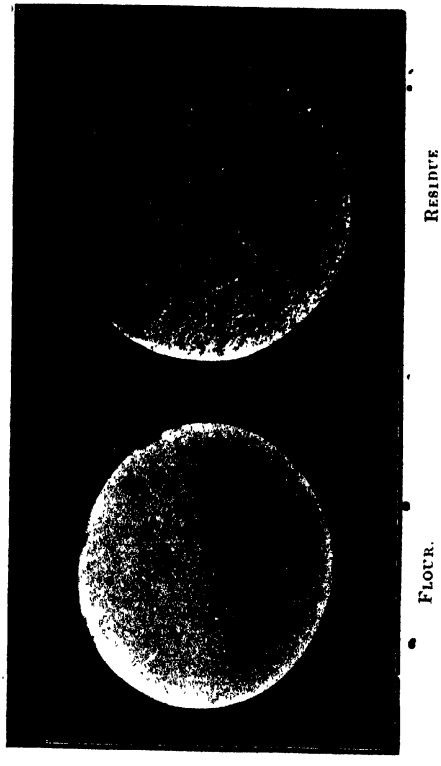
Now, Portland cement, as delivered by the manufacturer, consists of a mixture of coarse and fine particles, respectively known as **"residue"** and **"flour,"** and the value of a "floury" cement consists in this:—First, that the cementitious property of the material is believed to reside principally, if not wholly, in the very fine particles; and, secondly, that the "residue" consists of a practically inert material, or even possibly so much adulteration. This residue—when not an adulteration—is found to be a cement (generally the hardest and best burned material) that has been reduced, in the process of grinding, only sufficiently fine for the water added in use to attack the surface of the particles, the interior of which remains inactive. If these particles were reduced to flour, it would be found to be a good quality cement, showing a high tensile resistance (see Table B in this chapter).

Two samples of cement at present on the market, when tested for percentages of **residue, fine grit,** and **flour,** showed the following results:—

	Per cent.	Per cent.
Residue 180 by 180 sieve ...	12.3	9.20
Fine grit from Flourometer ...	27.5	35.66
Flour	60.2	55.14
	100.00	100.00

The ever-increasing fineness to which cement must be ground to meet modern demands has now, however, reached a point where ordinary methods of sifting are too inaccurate, and some other arrangement must be used for separating the finer particles into different sizes of grains.

It is true that fineness of grinding is not alone to be taken as indicating the quality of a cement—certain well-known statements to the contrary notwithstanding—as weakly



burned cements will give a larger proportion of fine material for the same amount of grinding than those with a harder clinker, though the latter are usually the more valuable as cement.

A good brand of Portland cement as ground at the present time should contain about 50 to 60 per cent. of impalpable flour which, it has been stated, is alone the active agency, and full size photographs are here given of test pats of the residue and flour of which a Portland cement is composed.

The proportions of the fine and coarse particles of Portland cement are shown in fifteen samples of cement which were tested for fineness on varying sieves and for flour percentage, and the results of these tests are as follows :—

Sample No.	% Residue 50 by 50	% Residue 76 by 76	% Residue 100 by 100	% Residue 180 by 180	% Flour in Flouro-meter
1	Nil	1.0	2.5	14.0	62.7
2	,,	0.5	2.5	14.0	60.5
3	,,	Trace	2.0	12.0	58.1
4	,,	,,	2.5	13.5	57.9
5	,,	0.5	2.5	13.0	65.5
6	,,	0.5	2.5	13.5	65.0
7	,,	0.5	2.5	13.0	55.0
8	,,	0.5	2.0	12.0	49.8
9	,,	0.5	2.0	13.0	59.0
10	,,	Trace	2.0	12.0	55.0
11	,,	0.5	2.0	11.5	51.0
12	,,	0.5	2.0	11.0	63.0
13	Trace	1.0	2.5	11.5	53.0
14	Nil	0.5	2.5	11.5	66.0
15	,,	1.0	3.0	12.5	55.0

Further, to show the actual adhesive and cohesive qualities of the fine and coarse particles of which Portland cement is composed, some grinding tests are here given showing the neat tensile strains of a cement as delivered, and the residue and flour tests of the same cement.

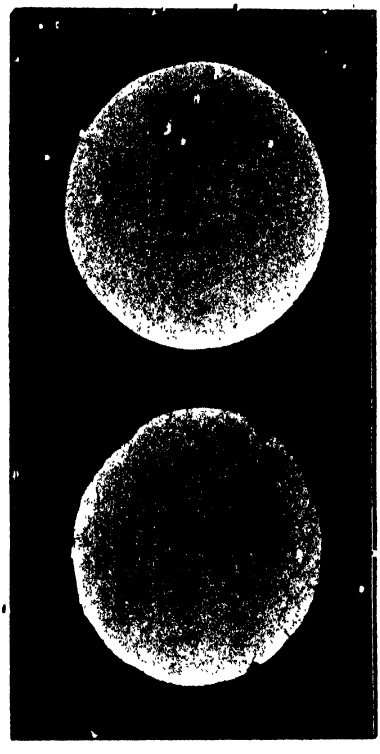
TENSILE TESTS OF NEAT CEMENT, FLOUR AND RESIDUES.

Description of Sample	7 days	28 days	Remarks
Cement as Drawn	740	890	Neat Cement, gauged
	750	850	with 18% water.
	700	900	
Average in lbs. per sq. inch ...	730	880	
Residue on 100 by 100 sieve ...	30	50	Neat Cement, gauged
	35	50	with 10% water.
	40	30	
Average in lbs. per sq. inch ...	35	43.3	
Residue on 180 by 180 sieve ...	70	110	Neat Cement, gauged
	70	110	with 10% water.
	80	110	
Average in lbs. per sq. inch ...	73.3	110	
Cement passed through 180 by 180 sieve	640	760	Neat Cement, gauged
	715	760	with 18% water.
	780	810	
Average in lbs. per sq. inch ...	711	776	
Flour from same Cement ...	520	600	Neat Cement, gauged
	525	635	with 30% water.
	550	645	
Average in lbs. per sq. inch ...	532	627	

The conditions resulting from the fine grinding of cement may be stated as follows :—

(1) A fine cement is used with greater safety in work. This is confirmed by the fact that the material is nearly always able to withstand the hot tests for soundness, and this again is due to the fact that the atmospheric moisture —or even the water used in gauging—at once attacks any

uncombined lime, and thus renders ultimate disintegration practically impossible. A photograph is here given of a coarsely-ground cement (15 per cent. residue 76 by 76 sieve) which has been subjected to a steam test and may



THE SAME CEMENT RE-GROUND AND SUBJECTED TO STEAM TEST.

A COARSE CEMENT SUBJECTED TO STEAM TEST.

be compared with a test pat of the same sample of cement when re-ground so as to pass through a 76 by 76 sieve. It will be noticed that the coarse sample shows a serious disintegration under heat, whereas the finely-ground sample

has withstood the same test without showing any sign of unsoundness.

(2) The fine grinding of a cement renders the material of a **quick-setting** nature (this, however, should not be confounded with what is ignorantly termed "hot" cement); the cause of this quick hardening being that the water used in gauging acts more readily on the soluble portions of the material now laid bare by the finer grinding. It must be noted, however, that finely-ground cements have a common tendency to develop **contraction cracks** when made up into test pats unless special precautions are adopted to prevent them, owing to the excessive proportion of water required to make a paste. To illustrate this point further, three photographs are here given of test pats of a finely-ground cement containing 1 per cent. residue on a 76 by 76 mesh sieve and 53 per cent. of flour. Plate 1 shows the neat cement pat which, when gauged, was enclosed in a damp box to set, and is thus thoroughly sound. Plate 2 shows another pat of the same cement placed in the air to dry, and which has developed some contraction cracks. Plate 3 is a quite sound test pat made up of a mixture of the same cement and sand in the proportions of one and one and treated under the same conditions as the unsound pat in Plate 2.

(3) Fine cement is found to develop its tensile strength in much shorter periods than a coarsely-ground product.

(4) One of the effects of the increased use of finely-ground cement is now very often seen in concrete work, and more particularly is this the case in the preparation of granitic or other floor construction work in concrete. There is no doubt that it is the aim of all those whose business it is to lay concrete floors to give an absolutely smooth and true surface. This is very pleasing to look upon, and credit is generally given to the pavior who gives the best and truest finish, but the frequent result is that the upper surface of these floors easily wears away, and causes a considerable amount of "sanding" or "**dusting.**" In these days of finely-ground cement, it is unwise to do so much surface trowelling, for this is undoubtedly the cause of the defect. If the granitic surface is once trowelled, then an excellent wearing surface results, and the upper layer of concrete in the granolithic will be

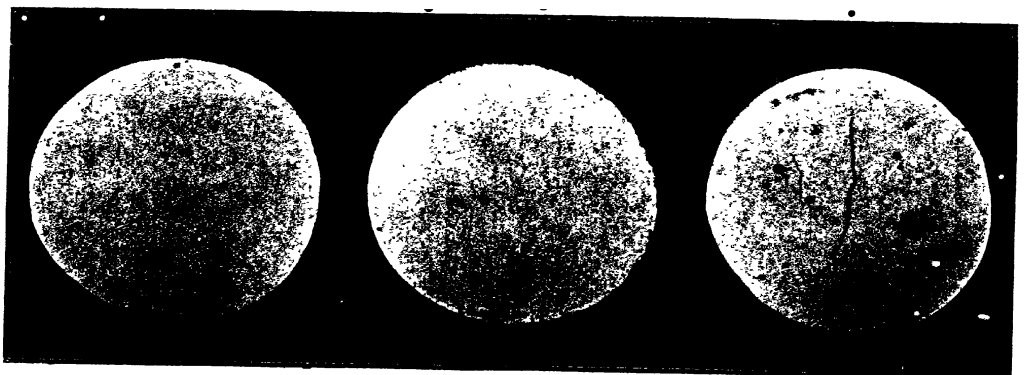


PLATE 1. PLATE 3 PLATE 2.

from 20 to 30 per cent. stronger. In the case of the setting of a finely-ground cement being interfered with by over-trowelling—which, by the way, was almost impossible with the old coarsely-ground cement—then a "dusting" surface is bound to result.

In considering the neat tensile strength tests of a finely-ground cement, therefore, it will be found that there are only slight differences between the three and seven days' tests. This is the result of the active particles being the more readily and immediately attacked after the gauging of the briquettes.

The following schedule of tests shows the neat and sand tensile strength results of three cements ground to varying degrees of fineness, viz. :—

TABLE A.

	a	b	c
Percentage of Cement passing through 180 by 180 mesh sieve	80%	85%	90%
	lbs. per square inch		
Tensile strength at 7 days, neat .. .	650	580	485
Ditto, at 28 days	725	650	545
Ditto, at six months	790	720	740
3 to 1 Sand and Cement at 7 days .. .	220	265	320
Ditto, at 28 days	250	320	380
Ditto, at six months	330	480	480

Other results as to the effects of fine grinding on cement are here tabulated.

TABLE B.—28 DAYS' TESTS. SAND AND CEMENT 3 AND 1.

With cement as received	221.5 lbs. per sq. inch
Same Cement re-ground to pass through 100 mesh sieve	422.5 " "
Same Cement re-ground to pass through 180 mesh sieve	465.0 " "
Cement flour	491.0 " "
Flour from re-ground residue	581.5 " "

These experiments were, however, made with an exceptionally poor cement, which, when received, contained only 28.00 per cent. of flour, the residue on a 76 by 76 mesh sieve being 10 per cent. and on a 100 by 100 mesh sieve 19 per cent. The sand used in the tests was made from Portland stone, and had been passed through a 20 by 20 mesh sieve and retained on a 30 by 30 sieve.

The value of a good quality modern cement (Table C.) containing 58 per cent. of "flour" may be here demonstrated by comparing the result of its tensile strength tests with those of other well-known brands of English cement (Table D.), but more coarsely ground.

TABLE C.

Fineness	Flour	3 Sand to 1 Cement, 1 in. by 1 in. section		
		7 days	14 days	28 days
5% 100²	58%	310	320	370
		310	320	360
½% 76²	,,	300	310	350
		300	310	350
Nil 50²	,,	290	300	340
		290	280	300
	Average	300 lbs.	306¾ lbs.	345 lbs.

TABLE D.

Fineness	Flour	3 Sand to 1 Cement, 1 in. by 1 in. section	
		7 days	28 days
14% 100²	42%	187 lbs.	245 lbs.
8% 76²	,,	183 ,,	276 ,,
4% 50²	,,	240 ,,	297 ,,
	Average	203½ lbs.	272⅔ lbs.

Again, the following are some tests of another sample of cement 76 per cent. of which passed through a 200 by

200 mesh sieve. The briquettes were made of one part cement and three parts standard sand by weight, viz. :—

	lbs. per square inch 7 days	28 days
Cement as taken from bag	343 ...	460
Residue on 200 mesh sieve	29 ...	110
Cement passed through 200 mesh sieve	489 ...	561

It will thus be seen from the results of these tests that the coarser particles of clinker absorb water very tardily and are practically inert and have very feeble cementing properties.

As to the definite action and results of high percentages of the coarser particles in cements there seem to be many theories, but little knowledge is available at present, since conditions vary to such extremes.

There is no doubt that the character of cement residues is governed entirely by the methods and materials and processes used in the manufacture of each identical sample; and further, it cannot be said at what point of fineness grains of cement begin to have a cementitious value, but it must be conceded that the coarser particles in a cement slake far more slowly than the cement flour. With the use of fine cements, therefore, there is the advantage that expansion will occur early in the process of hardening if it is to occur at all.

The ground product from a rotary kiln may be divided into (*a*) flour; (*b*) comparatively large pieces of clinker grit retained on the 180 by 180 sieve; and (*c*) fine particles, small enough to pass through the 180, but which are, nevertheless, particles of hard clinker grit. It is shown by tests that the **fine grit** (*c*) gives high expansion figures, whilst both flour and residue give rise to only a very slight divergence of the needles of the Le Chatelier mould.

The changes under the influence of boiling water thus observed in the three different stages of grinding are as follows :—

(*a*) At the time of gauging the cement the particles of flour, so-called, are hydrated more or less completely.

(*b*) Particles of clinker grit of comparatively large size

are not hydrated, except perhaps on the surface, and act throughout as inert material.

(*c*) Particles of fine grit (clinker), which are small enough to pass the 180 by 180 mesh sieve, are only partially hydrated at the time of gauging. The cement then sets hard, but under the influence of heat, when the block is boiled, the hydration of these small particles continues, with consequent expansion, unless the composition and burning of each particle are perfect.

In a test made on a portion of the sample from which the fine grit was taken, including the whole of the residue on the 180 sieve, it was shown that the original cement, boiled for six hours, expanded 29 mm. The cement minus the fine grit expanded 5 mm., and after seven days' immersion in cold water the expansion was 8.5 mm.

But to imagine that such an experience as related above would be found to be the case in all cements is quite a fallacy, and as before stated, such results may not be confirmed by further experiments with an average number of samples of rotary and other cements made by other methods and other machinery. An instance of this uncertainty in the results due to the expansion of the "fine grit" particles in cement (and from further tests carried out on the lines of the before-mentioned experiments) is shown in the following tests of three other samples of rotary kiln cement. The results then are quite contrary to those previously referred to, and from these results neither the difference in fineness of grinding nor the aeration of the cements seems to make any marked difference in the expansion as shown by the Le Chatelier method of testing for soundness. The samples of rotary cement here referred to were laid out $\frac{1}{4}$ in. to $\frac{1}{2}$ in. thick for aeration, and tested at varying periods, with the stated results.

It will be apparent, therefore, that although in some cements it may be found that the "fine grit" is a dangerous and expensive element, with age, yet in other cements both from rotary and stationary kilns the fine grit particles have no disturbing element at all after aeration; so it must again be said that the function of the coarser

AERATION RESULTS OF ROTARY CEMENTS BY THE LE CHATELIER TEST FOR EXPANSION.

Sample	Residue 180 × 180	Fresh		24 Hrs.		2 Days aerated		6 Days aerated		12 Days aerated		14 Days aerated		28 Days aerated	
		24 Hrs. in water	6 Hrs. Boiling	24 Hrs in water	6 Hrs. Boiling	24 Hrs. in water	6 Hrs. Boiling	24 Hrs. in water	6 Hrs. Boiling	24 Hrs. in water	6 Hrs. Boiling	24 Hrs. in water	6 Hrs. Boiling	24 Hrs. in water	6 Hrs. Boiling
		mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.	mm.
A	21%	Nil	2	.5	1.5	1.0	1.5	1.0	2.0	1.0	1.0	.5	1.5	1	.5
B	12%	1.0	2.25	.5	1.5	1.0	2.0	Nil	1.5	1.0	2.0	1.0	1.0	Nil	Nil
C	3%	Nil	2.0	.75	1.5	1.0	2.0	1.0	2.0	2.0	1.0	.5	2.0	1.0	.5

particles in a cement is determined more by the conditions and methods of manufacture in the product under notice than to any certain and definite result which is to be expected from all cements of whatever manufacture or process.

In a recent investigation relative to the influence of varying proportions of fine material in Portland cement, the most striking results were those in which the effects of normal cements with 17 per cent. and 2 per cent. residue respectively on a 180 × 180 sieve were compared. The results below are the average of five tests of each material, the briquettes being composed of one part of cement, 3 parts of normal sand, 2 per cent. of plaster of Paris, and 8 per cent. of water.

STRENGTH IN LBS. PER SQUARE INCH

Residue on 180 sieve	7 days	28 days	3 months	1 year
17 per cent. ...	375.9	437.4	529.7	566.0
2 per cent. ...	416.0	485.6	576.5	617.7

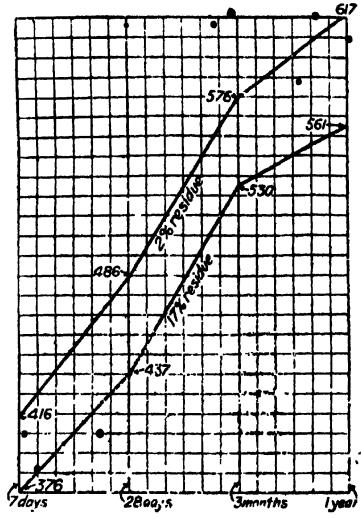
The accompanying curve illustrates these results, and shows still more clearly the benefits to be obtained by grinding the cement so as to leave as small a residue as possible on the 180 by 180 mesh sieve, which is at present the standard sieve for the testing of the fineness of Portland cement.

Where a neat cement for any detail of constructional work is used, the same results as would be obtained with a coarsely ground material must not be expected from a modern finely ground product; and since a good proportion of residue is now done away with in first-class cements a mixture of sand may be necessary to ensure against the contraction cracks which would probably develop in a fine cement if used neat.

The possibilities of economy and increased efficiency in

the fine grinding of cements are by no means sufficiently appreciated at the present time by cement users; and it is not overstating the case to say that by using a finely ground cement, with a judicious admixture of sand, the contractor's work can be carried out far more satisfactorily, and at less cost than with coarser cements.

It has been shown, then, that the proportion of flour



Tensile Strength Tests of Cement at a Varying Fineness.

and residue contained in a cement is a point of material importance; and after the percentage of each has been ascertained in a given sample an intelligent as well as an economical use of the material should follow from a knowledge of the manufacturer's increased expense in production, and the reduction of the works' output in supplying a finely ground product.

Thus from what has been stated, it will be seen that the test for fineness should be directed to determining the proportion which is very fine rather than the residue above a certain size, and this points to the way in which the fineness test should be carried out.

While it is generally recognised that the finer cement is ground the greater is its cementitious value (the coarser particles having little or no setting property according to their respective sizes), all the researches which have been made in connection with the fineness of Portland cement convey very little information with reference to the effect of fineness of grinding upon the strength of concrete. This deficiency has recently been made up in some measure by the results of five series of tests conducted for the American Portland Cement Association at the Lewis Institute, Chicago.

Covering a period of over four years, the investigation comprised some 6,000 compression tests of concrete, 9,000 compression and tension tests of mortar and several thousand miscellaneous tests, fifty-one different samples of cement being used, ranging from 4 to 7 degrees of fineness. It is evident, therefore, that the results of the tests afford an adequate basis for the drawing of reliable conclusions, chief among which are the following :—

(1) There is not necessarily any relation between the fineness of the cement and the strength of the concrete, if different qualities of cement are considered.

(2) As a general rule, the strength of the concrete increases with the fineness of a given lot of cement, for all mixtures, consistencies, gradings of the aggregates and ages of the concrete.

(3) Cements with residues lower than 10 per cent. are inclined to give erratic results in the strength tests, but for residues higher than 10 per cent., the strength of the concrete varied approximately in inverse proportion to the residue on the 200 mesh sieve.

(4) Finely ground cement is more effective in increasing the strength of poor rather than rich mixtures.

(5) Fine grinding is more effective in increasing the strength of concrete at 7 days than at from 28 days to a year.

(6) The decreased benefit of fine grinding with the age of the concrete does not bear out accepted opinion that the coarser particles of cement do not hydrate, but indicates that the principal result of finer grinding is to hasten the early hardening of the concrete.

(7) For the richer mixtures and the consistency necessary for building construction, the fineness of the cement has no appreciable effect on the workability of the concrete.

(8) The normal consistency of concrete increases with the fineness of the cement, and about 0.1 per cent. of water (in terms of the weight of the cement) must be added for each 1 per cent. reduction in residue on the 200 mesh sieve.

These results of the experiments support the view that the ultra fine grinding of cement is a feature that has few real or permanent advantages. Fine grinding increases the strength of concrete at early dates, but the merits of fine grinding at periods exceeding a month are not so pronounced and the constructional engineer is more concerned with the strength of the concrete at ages of two months and onwards when he applies the full load to his work.

Chemical composition has a greater effect upon quality than fineness of grinding, *e.g.*, one brand of cement with a raw residue of 15 per cent., and cement residue of 13 per cent., is found to be as good in quality, or better, than another cement with raw and cement residues of 4 per cent. on the 180 sieve.

It has been generally accepted in this country that "the finer the cement the greater its strength when

mixed with sand and aggregate," and this belief has received support from results of investigations recently made by the American Society for Testing Materials.

Starting from a basis of cement having 20 per cent. residue on the 200 × 200 mesh sieve, and upon investigating the compressive strength of concrete mixtures varying from 6 parts of aggregate to 9 parts of aggregate per part of cement, it has been established that for each degree of improved fineness the following increases of strength occur :—

7 days	-	2.3 per cent.
28 ,,	-	1.8 ,,
90 ,,	-	1.4 ,,
180 ,,	-	1.1 ,,
360 ,,	-	1.0 ,,

Thus, concrete containing cement with a residue of 15 per cent. on the 200 mesh sieve is 5 per cent. stronger at a year than similar concrete containing similar cement with a residue of 20 per cent. on the 200 mesh sieve, and at an age of 7 days the concrete is 11.5 per cent. stronger.

There would, of course, be a limit to this ratio of increase in strength, and the calculation is, therefore, no doubt, intended to apply to proportionately small increases in the fineness of grinding above the standard given.

Reverting to the **fineness test,** the sample of cement for sieving should be a quantity weighing 100 grams as perhaps the most convenient. The weight in grams of the residue then remaining on the sieve represents the percentage retained by that particular sieve. A convenient size sieve for this test is about 8 in. in diameter. The manner in which the shaking is carried out determines the length of time required for the operation ; some 15 minutes' gentle and continuous tapping of the sieve on a table should, however, be found ample.

The **sieves** used for the test should be frequently examined, under a magnifying glass if possible, to see that

no wires are displaced, leaving apertures larger than the normal.

This system of determining the fineness of Portland cement by the percentage of residue on a certain sieve has been subjected to much enquiry and argument, chiefly on account of the variations found in the thickness of wire of which the gauge of the sieving is manufactured.

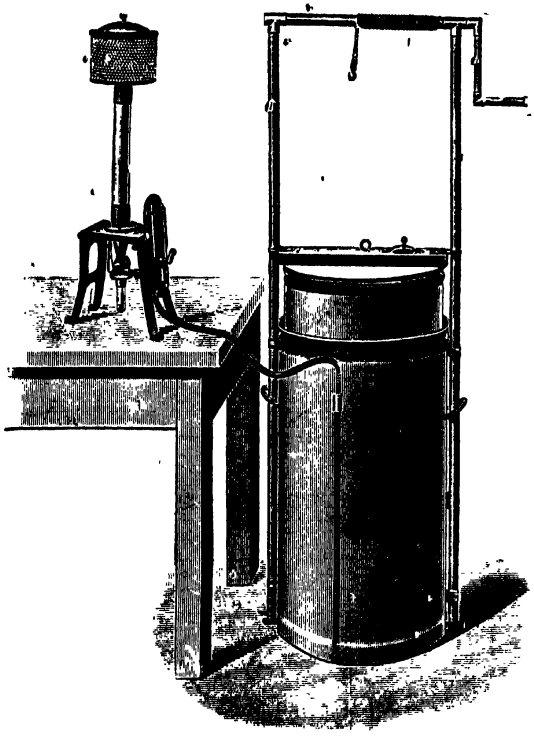


Cement Testing Sieves.

In this country there had been no general standard of size for the wire until the British Standard Specification gave such details, nor was there any agreement as to how long the actual sieving process was to be continued. This has, however, now been clearly defined, and the importance of the thickness of the wire used in sieve-making is obvious when the difference is observed in the results of the tests for fineness by two sieves of the same number of meshes per square inch, but which differ materially in the size of the wire; the reason, of course, being that the apertures are much smaller in one case than in the other.

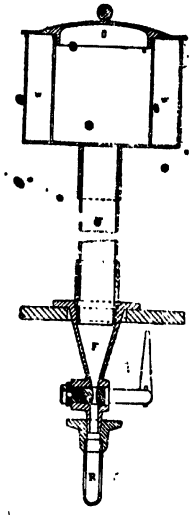
Since, however, it is desirable to know the percentage of actual **flour** in a given sample, according to the different classes of grinding machines used in the process of manufacture, an apparatus has recently been devised for this purpose, and is illustrated here. The machine was designed with a view to putting the estimation of the flour content on a "quantitative" basis, and is termed the **Flourometer.** The principle of this instrument is simple. A definite amount of cement is "washed" free

from "flour" by a current of air, and the non-flour residue is weighed; the difference between the original



THE STANDARD FLOBROMETER FOR THE ESTIMATION OF THE "FLOUR" IN CEMENT.

and the final weights giving the proportion of the flour present.



The Flourometer has thus been designed to overcome some inconveniences found during the practical working of the test with an earlier flourometer apparatus. The new pattern may be said to be almost automatic; a weighed sample of cement having been placed in the instrument (F), the bell of the aerometer is raised to its full extent, a stopcock is opened and the apparatus can then be left without attention. In ten minutes time the separation of the flour will be completed, and the residue will be found collected in a small removable glass receptacle (R). It then only remains to weigh the residue to determine the percentage of flour contained in the cement. To make another test it is necessary to replace the residue receptacle and proceed as before; there are no screws or washers to be fitted and arranged as in the old pattern previously introduced, and in consequence far more tests can be made in a given time.

Repeated tests with this Flourometer have conclusively proved that its results are more accurate than those of the original pattern; further, there is the saving of time, ten minutes against three-quarters of an hour per test, the elimination of the need for constant attention, and the ease with which tests can be made rapidly one after another—points which will find greater appreciation than resulted from the introduction of the first Flourometer.

For ensuring the economical working of Griffin, Ball and Tube, and other fine-grinding mills an appliance of this kind is valuable, as it enables tests to be made at frequent intervals, and indicates how the mill may be adjusted so as to give a maximum of powder of the required fineness with the expenditure of a minimum of power.

Tests for Bushel Weight and Specific Gravity.

CHAPTER XIX.

THE present period of the fine grinding of cement has rendered useless and made obsolete the old-time tests of "weight per bushel." So long as all cements were ground to about the same degree of fineness—a degree which, by the way, was not to be compared with the fineness of cement now put on the market—the requirement that a cement should weigh a specified number of pounds per striked bushel was found fairly satisfactory when made workable by uniform methods in carrying out the test. In some specifications even now one may find it stipulated that the cement shall weigh "not less than 112 lbs. per striked bushel," and qualifying details of the test are added as to the way in which the measure shall be filled, viz., from a hopper about 18 inches above.

The weight of cement per bushel was a test first used in the series of cement tests, and its object was doubtless to ensure the material being of a well-burned and unadulterated quality. The test was in vogue when what was presumed to be a heavy cement was more in favour than a light cement, without regard to fineness; but its continued use to-day would only be of value if the bushel tests were of a graduated scale, according to the fineness of the grinding, for the finer a cement is ground the more bulky it is, and the less is its weight per bushel. Contrary to the old experience, therefore, the **weight per bushel** of cement if demanded to-day should be of a reduced quantity. Rotary kiln cements are, however, generally and comparatively heavy in weight per bushel.

As the call for a finer cement increases, the weight of 112 to 120 lbs. per bushel, once easily attained cannot now be reached, and the weight of a well-burned ordinary

cement ground fine enough to pass through a sieve having 5,776 meshes to the square inch (76 by 76) will be about 105 to 110 lbs., and 110 to 115 lbs. if a Rotary cement.

The weight per bushel test is now sometimes used by contractors who are in the habit of employing cement by measure, and they thus desire as bulky a product as possible, providing its tests in other ways are good. The weight test may, therefore, still have its sphere of utility. When the test is used, therefore, it must be carried out with care, as there is a considerable personal factor to be reckoned with. The old methods of determining the weight of cement per bushel consisted of apparatus so arranged that the hoppers have no direct communication with the bushel measure. The cement thus falls into the measure at a uniform rate and to an equal density, affording comparative determination between the weights of different brands of cement.

The quality shown in checking the weight of cement was later, however, generally ascertained by calculating its **specific gravity,** or the weight of cement as compared with the weight of an equal volume of water or other standard substance at the same temperature and pressure. This test is carried out with facility by one or other of the simple appliances which have been devised, and the results are immediately known.

The specific gravity of Portland cement which has not been calcined at a sufficiently high temperature, or which contains a considerable portion of free lime, varies from 2.9 to 3.05, and a good cement when freshly ground usually has a specific gravity exceeding 3.10. The age of the cement to be tested must first be taken into account in appreciating the value of this test, the results of which are found to decrease according to the time the cement has been in store, because of its absorbing carbon dioxide and water vapour from the atmosphere. Most freshly-ground cements have a specific gravity above 3.10 and an aerated cement can be brought back to its orginal condition by heating to redness for a few minutes, and so driving off the moisture and carbonic acid gas absorbed since being ground.

A high specific gravity does not, however, necessarily prove a cement to be of high quality; for an adulteration

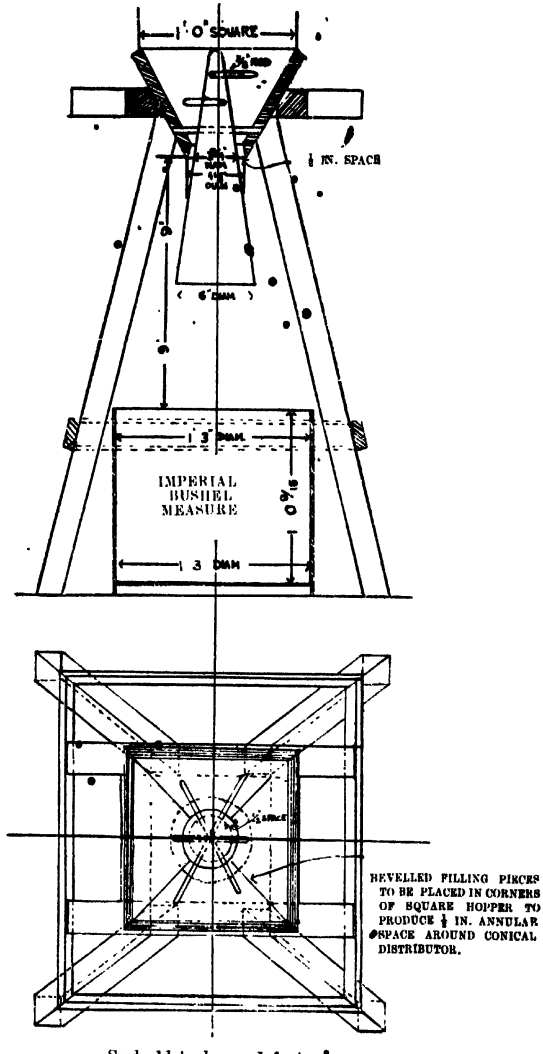
may be added, such as slag, which does not appreciably reduce the specific gravity. Generally speaking, however, the specific gravity test has a value in that other adulterations most likely to be practised, and most to be feared, are made with materials which reduce the specific gravity; and the test is also said to be sometimes useful in determining an under-burned cement which may fall below 3.00, or an over-burned product. The test for specific gravity is of value when care is exercised in the ascertaining of results, and although rarely adopted as an ordinary test by cement users, yet with the accuracy of the laboratory, where the necessary appliances are at hand, a reliable result is readily obtained.

The chief causes likely to affect test results, and, therefore, to be guarded against, are as follows :—

Incorrect weighing of the cement sample for test.
Variation of temperature during test.
Waste of cement in filling apparatus.
Air bubbles.

Some authorities find that owing to absorption of water and carbonic acid (having specific gravities of 1.0 and 0.88 respectively), the specific gravity of cement decreases with age, or after aeration, but it is surprising that, after ignition at red heat to expel the water and carbonic acid and to reduce the material to practically the same condition as regards these substances as when it left the kiln, the specific gravities of various cements were so nearly identical as to render the test of little or no value as an indication of quality. The results of preliminary experiments with thirty different cements, having specific gravities varying between 3.026 and 3.138—*i.e.*, a difference of 0.112—showed that after ignition the specific gravities differed by only 0.016, which is well within the range of experimental error in ordinary technical determination.

In order to test the generally accepted theory that specific gravity is an indication of the degree of calcination, twenty-eight samples of black well-burned, and yellow under-burned clinker from the same kiln or charge were obtained by Mr. D. B. Butler from various works, and the specific gravity of each sample was ascertained (1) in the condition in which it was received, and (2) after ignition at red heat. The results showed that all the


1'0" SQUARE
¼ IN. SPACE
IMPERIAL
BUSHEL
MEASURE
BEVELLED FILLING PIECES TO BE PLACED IN CORNERS OF SQUARE HOPPER TO PRODUCE ¼ IN. ANNULAR SPACE AROUND CONICAL DISTRIBUTOR.

Scale 1½ inches = 1 foot.

HOPPER AND BUSHEL MEASURE FOR ASCERTAINING WEIGHT OF PORTLAND CEMENT.

yellow under-burned samples, when they had absorbed any appreciable amount of water and carbonic acid, had a much lower specific gravity than the well-burned samples, but that after ignition at red heat the specific gravities of the well-burned and under-burned materials were practically identical.

The specific gravity clause is no longer included in the British Standard Specification for cement, and a laboratory can probably make no greater mistake than to reject a cement on the ground of low specific gravity alone unless it has been ascertained that this low gravity will actually affect the quality of the work. An abnormally low specific gravity in cements is caused by the presence of water combined or absorbed, by under-burning, or by adulteration or blending. Now where a low test is found it should be at once the work of the laboratory to discover the cause of this condition and not to condemn the cement outright for a quality that may be actually beneficial.

It is well known that the absorption of moisture during the process of seasoning will cause a considerable lowering of the specific gravity.

Another series of tests was recently made on this point by taking samples from bags of cement which were exposed to the ordinary air of the laboratory and making weekly determinations of the specific gravity and monthly determinations of the strength. The cement as recorded was comparatively fresh, and gave a gravity of 3.12 as it stood and 3.17 after drying at 212 degrees F. At six months it gave 3.09 undried and 3.16 dried, in one year 3.06 undried and 3.14 dried, and at two years 3.00 undried and 3.09 dried. At all periods the values from the ignited cement were practically the same. The strength tests on these cements were satisfactory up to one year, after which time they ran rather low, the best tests—showing the most satisfactory increase in the curves—being those of six months, although at this point the specific gravity was below specification.

The first esential, therefore, on finding a low gravity, is to ascertain by drying, igniting, and by moisture determinations how much of it is due to the ageing or seasoning of the material. If such is found to be the cause, and at the same time the strength tests are satisfactory, there

certainly is no reason for rejecting a material probably safer than the average.

If the low specific gravity is not caused by ageing, it should be then examined for adulterations, and if not adulterated, it is probably due to under-burning, which, if existing sufficiently to cause unsoundness, should make the material unfit for use. If the results of the specific gravity test are thus investigated the test becomes one of considerable interest; a single determination, however, without further study is almost valueless.

Other cement testers have given a very careful study to the utility of the specific gravity test of Portland cement, and it has been maintained that the test is of no value whatever in detecting under-burning, as under-burned cement will sometimes show a specific gravity much higher than is set by the standard specifications. Under-burned cement is, however, readily and promptly detected by the soundness tests, and no others are needed for this purpose.

Again, the value of the specific gravity test as an indication of adulteration has been much exaggerated, for while a large admixture of any light adulterant with the cement would be shown, there is at the same time much slag and also other foreign materials which could be mixed with cement in large quantities without lowering the specific gravity below the limit previously allowed by the standard specification.

It then appears that low specific gravity is usually caused by seasoning the cement or the clinker, either of which improves the product, and again the proposition to ignite the cement sample which falls below specification and determine the specific gravity upon the ignited portion is of little value, because adulterated cements also have their specific gravity very much raised by such ignition.

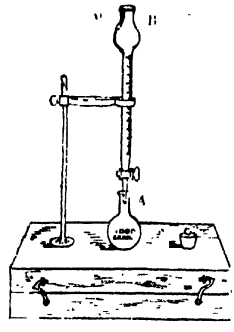
Too much importance must not, therefore, be attached to the specific gravity test and results, and before condemning cements under this test an intelligent investigation is first necessary as to the cause of failure to come up to the prescribed weight.

A large number of **appliances** have been devised for

carrying out the specific gravity test, and a brief description, together with illustrations of those more commonly in use in this country, is given here.

MANN'S GRAVIMETER.

This apparatus, one of the first used for the test, consists of a small glass beaker, which is to be filled with 1,000 grains of cement.



MANN'S GRAVIMETER.

The graduated pipette held above is filled to the mark B with paraffin—this being the cheapest material which does not act on the cement—and the paraffin is then filled into the lower vessel until the contents reach the mark A.

The height of the column of liquid remaining in the pipette determines the specific gravity, which can be at once read off the stem, the size and graduations of which are specially prepared for this test.

KEATES' SPECIFIC GRAVITY BOTTLE.

The ordinary specific gravity bottle affords as correct a result as can be wished, but necessitates a knowledge of the specific gravity of the liquid employed for the test.

Here the upper bulb of the apparatus is of known capacity (1,000 grains), and the vessel is filled with liquid (generally turpentine) to the mark A and weighed.

The cement is then introduced until the level of the liquid reaches B, when it is weighed again.

The difference of weighing is added cement, and the specific gravity is calculated thus :—

The capacity of the top bulb between marks A and B is 1,000 grains water measure, which equals 64.8 cubic centimetres, *i.e.*, 64.8 grammes of water.

Hence the specific gravity =

$$\frac{\text{Weight of added cement in grammes.}}{64.8}$$

In this case the volume is fixed and the weight variable, but in the following method of test the weight is fixed and the volume variable.

BLOUNT'S VOLUMENOMETER.

An extremely simple form of the specific gravity tester is as named above, and consists of a small flask, flattened so as to ensure its stability, and provided with a short slender neck with fine graduations. The lowest graduation mark is 14 c.c. (the reason for which will be seen presently), and the capacity of the flask up to this mark is known, and equals 64 c.c.

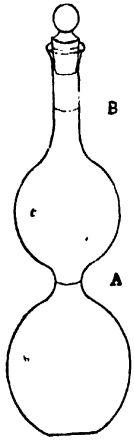
The instrument is used by running into it 50 c.c. of oil of turpentine from a pipette, which has been accurately calibrated against the flask. As the oil of turpentine is introduced by means of a pipette, the neck of the apparatus is kept dry. A known weight of cement (50 grains) is then added through a small funnel so as not to block the tube. This cement, of course, displaces a certain volume of the liquid and raises its level in the bottle; and as all ordinary cements have a specific gravity such that the displacement caused by 50 grains falls between 14 c.c. and 20 c.c., the former has been taken as the lowest graduation mark on the neck. Any displacement

greater than 14 c.c. is therefore registered on the neck, and may be at once read off.

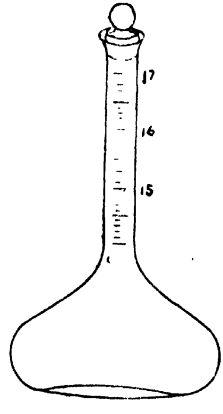
$$\text{The specific gravity} = \frac{50\ (i.e.,\ \text{weight of cement})}{\text{Volume of liquid displaced, in c.c.}\ (i.e.,\ \text{reading on the neck})}.$$

The temperature of the turpentine must be known, but need not be fixed at any standard point, and after the operation the volumenometer (which is stoppered to prevent evaporation) is brought to the same temperature by immersion in the same vessel of water as that previously used for the stock bottle of turpentine.

Seeing that in all cases the temperature before and after the displacement of the liquid by the cement must



KEATES' SPECIFIC GRAVITY BOTTLE.



BLOUNT'S VOLUMENOMETER.

be identical, because of the high co-efficient of expansion of oil of turpentine and petroleum, it is evident that an apparatus of small total capacity, such as that described above, is convenient, as it can be immersed bodily in a

beaker of water and brought to the proper temperature with ease and rapidity.

GRIFFIN'S SPECIFIC GRAVITY FLASK.

This appliance is convenient in that a larger quantity of cement may be dealt with, and so the possibility of error experienced in using a small quantity of cement is obviated. The test is carried out by introducing 200 cubic cent. of petroleum into the dry flask, keeping the neck of the flask thoroughly dry so that the cement does not adhere when ultimately added; 150 grammes of cement are then carefully introduced.

The flask should then be stoppered and lightly shaken so as to remove any air bubbles; the contents are then allowed to settle in a vessel containing water of a temperature equal to the petroleum when added, and the cubic centimetres read off on the graduated stem.

In calculating the specific gravity, the 200 c.c. of petroleum should be deducted from the reading, and the result will give the volume occupied by 150 grammes of cement, thus: if the displacement is at 249.0 it follows that 150 grammes of cement have displaced 49 c.c. of petroleum. The displacement divided into the weight of cement (150 grammes) gives the specific gravity, which in this case would be 3.061.

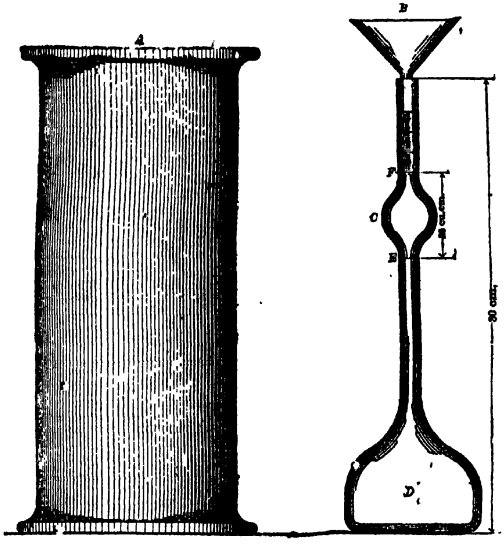
LE CHATELIER'S SPECIFIC GRAVITY TEST.

The determination of specific gravity is also conveniently made with Le Chatelier's apparatus. This consists of a flask (D) of 120 cu. cm. (7.87 cu. ins.) capacity, the neck of which is about 20 cm. (7.87 ins.) long; in the middle of this neck is a bulb (C) above and below which are two marks (F) and (E); the volume between these marks is 20 cu. cm. (1.22 cu. ins.). The neck has a diameter of about 9 mm. (0.35 in.) and is graduated into tenths of cubic centimetres above the mark (F).

Benzine (62 degrees Baumé naphtha), or kerosene free from water, should be used in making the determination.

The specific gravity can be determined in two ways:—

(1) The flask is filled with either of these liquids to the lower mark (E) and 61 gr. (2.25 oz.) of powder, previously dried at 100° Cent. (212° Fahr.) and cooled to the temperature of the liquid, is gradually introduced through the



Le Chatelier's Specific Gravity Apparatus.

funnel (B) [the stem of which extends into the flask to the top of the bulb (C)], until the upper mark (F) is reached. The difference in weight between the cement remaining and the original quantity (64 gr.) is the weight which has displaced 20 cu. cm.

(2) The whole quantity of the powder is introduced, and the level of the liquid rises to some division of the graduated neck. This reading plus 20 cu. cm. is the volume displaced by 64 gr. of the powder.

The specific gravity is then obtained from the formula:

$$\text{Specific gravity} = \frac{\text{Weight of cement}}{\text{Displaced Volume.}}$$

The flask during the operation is kept immersed in water in a jar (A), in order to avoid variations in the temperature of the liquid. The results should agree within 0.01.

A convenient method for cleaning the apparatus is as follows: The flask is inverted over a large vessel, preferably a glass jar, and shaken vertically until the liquid starts to flow freely; it is then held still in a vertical position until empty; the remaining traces of cement can be removed in a similar manner by pouring into the flask a small quantity of clean liquid and repeating the operation.

Setting Tests.

CHAPTER XX.

WHEN Portland cement is mixed with water, a plastic paste is formed which ultimately hardens into a stone-like substance, and according to the activity shown in this transition from a paste to a thoroughly hard mass, so is the material found to be of a quick or slow-setting nature.

It is of importance that the cement user should be aware of the setting properties of the cement to be employed in constructional work, for unexpected results are apt to ensue from the omission of this test.

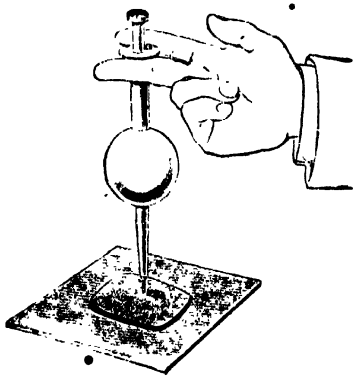
It often happens that when a quick-setting material is inadvertently delivered on to the contractor's works, and is brought into use, the setting has commenced even before the wet mixing of the aggregate and cement is completed, with the result that this setting action being unnoticed, is interfered with, and the material will not again set for some days, or, at least, until the moisture has thoroughly dried out of the mixture. Its cohesive value is often then so greatly reduced as to render the cement practically valueless. This is a common occurrence in the use of cement, and may easily be avoided by a systematic testing of its activity. Care should first be taken to ascertain the setting time of the cement before it is to be used, and next, that no greater bulk of mortar is mixed than can be used before the setting commences.

Under no circumstances must a cement which has once been allowed to set be re-tempered or re-mixed for use, since from what has been said, it will be seen that the material is thereby liable to be rendered worthless.

Cement manufacturers have it in their power to make either a quick or slow-setting cement, and unless other-

wise instructed supply the latter, which takes thirty minutes and upwards to harden on the surface of the test pat. A **quick-setting** cement commences to harden in a few minutes, and although perhaps equally strong in its tensile resistance and constructional value, yet a cement of this nature must be used with care. It is employed only for special purposes, such as in operations under water, in plaster work, and when working in cold weather. In all other cases it is most advantageous to use a slow-setting cement.

This is indeed indispensable when dealing with careless workmen not under supervision, who always prepare their



METHOD OF HOLDING NEEDLE IN TAKING THE SETTING TIMES OF CEMENT

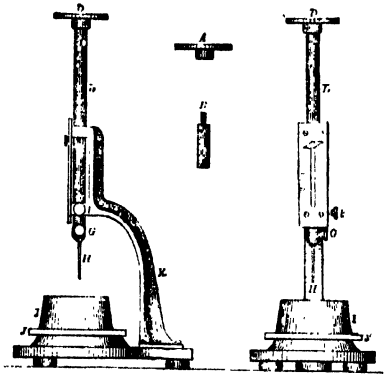
mortar many hours ahead and even sometimes "drown" it with excessive quantities of water.

Two distinct setting time tests are at present in use, viz., one for the commencement of the setting called the **initial** set, and the other for the end, or **final** set, the time when a weighted needle makes no impression on the test pat. This duplicating of the test is theoretically somewhat useless and superfluous, for the cement must, of course, harden, and if it did not the tensile strength or other tests mentioned would soon show the fact. Of these two tests

then the easier to record with accuracy is that for the initial setting, and with the Vicat needle now so widely used with this test, the initial set is easily taken and unmistakably observed.

However, the method chosen for testing the setting of cement matters little, provided that one definite test is adopted and that all its conditions are well defined in this respect.

The British Standard Specification originally arranged that a specially designed weighted needle should be used



THE VICAT NEEDLE.

for testing the setting, and the existing specification further states :—

Unless a specially quick-setting cement is specified or required, it shall have an initial setting time of not less than 20 minutes and a final setting time of not more than 10 hours.

If a specially quick-setting cement is specified or required, it shall have an initial setting time of not less than 2 minutes, and a final setting time of not more than 30 minutes.

The methods for determining the setting times of cement

are described in the Standard Specification, which is included in the appendix, and the standard test to-day is taken by the Vicat needle here illustrated.

It is well known that different brands of cement, indeed the same cements, when tested in different parts of the country, even by the same operators, require different percentages of water in gauging and have also widely different results, and to use a standard quantity of water for testing all cements in preparing the pat or sample for testing is, of course, impracticable. But even with the same percentage of water used in mixing and with merely a difference in climatic conditions, the results of the setting time test will vary considerably. Similarly the size and weight of the needle used and the quantity of cement employed in the test all affect the setting time results.

With the advent of the rotary kiln cement, the setting of cements has received much special consideration, and it has been found that where long-tried methods of regulating the setting times of cements made by the fixed kilns have been quite satisfactory, yet with the newer product—which, it may be added, is alone constituted from a fully burned clinker free from mixtures of under-and over-burned clinker and excessive fuel-ash and dust—many of the older methods of regulating the set have failed, and new arrangements have perforce been made with more or less satisfactory results.

In the early days of rotary cement one heard sometimes of these cements taking days and even weeks to harden off in concrete work, and this is often the case where the **flash set** of a cement has passed unnoticed, and then ultimate **hardening** in such a case is only equal to the drying of the cement or concrete, in the same way that the setting of an ordinary kiln cement is delayed considerably by interfering with the natural set.

Some cements when freshly made have this "flash" set, that is, as soon as water is added in preparing for the test, the paste stiffens, and, upon remixing, the initial and final sets are still readily perceptible in the usual way, but in this case the hardening of the cement or concrete takes a considerable time, as above stated. The ultimate results are, however, accompanied by more serious consequences

and perhaps a delay in the constructional work waiting for the hardening of the concrete.

It will thus be seen that in some cases the setting of a cement can be traced to a "flash" set, an "initial" and "final" set, and a "hardening"—all distinct and easily definable conditions and results which the rotary cement has brought in its train with its increasing manufacture and use in this country.

To regulate the setting of rotary kiln cement, therefore, it has been found that whereas a small percentage of **gypsum**—a mineral with calcium sulphate as its essential constituent, and occuring in various parts of the country—added to the product when cement grinding occurs, generally does all that is desirable and necessary in slowing down an inordinately quick-setting cement, yet to-day, the manufacturer has recourse to steam hydrating in the mills during the clinker grinding operation, or extensive "weathering" of the clinker before grinding, or the watering of the hot clinker when leaving the kilns. These methods of manufacture in slowing the set have been necessary where the addition of gypsum was found to be practically useless, but they cannot always be depended upon to give satisfactory results.

Gypsum is often more effective in its burnt state as **plaster of Paris** than in the raw state.

In this way the invariable quick-setting feature of rotary kiln cement is regulated to the setting times required in the marketable product.

Generally speaking, however, a cement when put upon the market, and after being mixed neat with water, should remain inert for at least one hour, and after that time the quicker it sets the better.

The addition of gypsum is generally recognised up to about 2 per cent., and the introduction of this material is, as explained, rendered necessary so as to regulate and make uniform the general setting tendencies of the cement in bulk.

For instance a cement clinker when ground from the kilns may vary in its setting time from a few minutes to some hours, and such a material to a consumer would prove exceedingly irksome and inefficient, but by the introduction of the gypsum which is mixed with the

material whilst passing through the grinding machinery, a cement is obtained which can be relied upon as being of a slow and generally uniform set.

The addition of gypsum on the other hand in no way interferes with the strength of the cement, and its introduction does not render careful manipulation before use any the less desirable.

With rotary kiln cement, the addition of gypsum to retard the set is sometimes of very little use. This is due to the lack of a little free lime in the clinker, without which gypsum is quite inert. In such cases, however, a mixture of 1 per cent. of gypsum and 1 per cent. of caustic lime has been found to regulate the setting.

Rotary cements are usually so well burnt that all the lime is in combination, and there is considerable difficulty in regulating the setting by any other means than those of wetting and storing the clinker for, very often, some one to three months.

This is indeed the practice in many works, and in such cases the cost of grinding is much reduced and the clinker will also probably be found to contain the necessary small amount of free lime to make the gypsum operative.

The watering or hydrating of well-burnt clinker to regulate the setting of cement is frequently employed, and is of advantage if storage and "sweating," as it is termed, follows.

The set generally becomes very slow under this process, and the finished cement can thus be regulated to any desired time of set by mixing the hydrated clinker with various proportions of new quick-setting clinker.

Occasionally, cements which are slow-setting when freshly ground, have been known to undergo conversion to quick-setting on storage, and then either retain that character or revert to the original state. This is probably due to one of two things: (1) the high percentage of alumina (especially when the cement is prepared by the dry process of manufacture), which necessitates the addition of gypsum. This latter is soluble, and retards the setting of the cement, but only for a time, for when the sulphate of lime enters into combination with the calcium aluminate its restraining action is removed and the cement

becomes quick-setting. (2) The calcium oxide, which is sometimes present in freshly-burned cement clinker, but which may be gradually converted into carbonate on storage. When this is the case, the cement is found to set more quickly after storing than when freshly ground. And in the event of using such a cement the hardening of the concrete is inordinately delayed if the cement set has been interfered with.

It is, then, generally understood, though not always the case, that a quick-setting cement is rendered of a slower-setting nature by storage, on account of the hydration of the calcium aluminate contained in it; but by this statement it should not be inferred, as it often is, that a quick-setting material is necessarily one which has not been stored, and is what is commonly termed "**hot**" or "fresh." These terms are undoubtedly very misleading to the average cement user. The fact of its being freshly ground has little or nothing to do with the setting properties of Portland cement; its chemical composition alone is responsible and accountable for the difference in the times of setting.

As the *strength* of a cement is known to be due to the effects of the calcium silicate contained in the material, so is its *activity* said to be due to the calcium aluminate.

Portland cement usually contains between 4 and 8 per cent. alumina, and other things being equal, the rapidity of setting increases in proportion as the alumina rises above 6 or 8 per cent., so it is held that a material which is shown by its chemical analysis to contain more than this amount of alumina sets very rapidly indeed.

There are some strict conditions which must be observed in testing the activity of cements. One of these is referred to more particularly in the preceding article bearing on the results of fine grinding, which more often converts into a cement of quick action what would otherwise be a slow-setting material. Again, the temperature of the test-room has a serious influence on the speed of setting—a high temperature causing more rapid action. Sufficient uniformity of temperature will result, however, if the test-room be kept comfortably warmed in winter, and in summer if the specimens be kept until set under a damp cloth out of the sun in a cool room.

Generally speaking, also, both the initial and final periods of setting are prolonged by an increase of water in mixing, and they are again affected by the different climatic conditions under which the test is made. The different judgment of observers also makes the test somewhat erratic and variable.

Then in addition to the checking of the setting times of cements, the variableness in the **final hardening** of different brands of cements is an important factor to be inquired into, but this point is more particularly referred to at the end of this chapter.

By means of a microscope and with a power of about 200, it is possible to watch cement in the process of setting, and for this purpose a little cement should be dusted on to the slide and fastened down by a cover-glass, and both placed under water.

It will then be found that the grains of cement become covered with crystals, which make their appearance sometimes in from ten to twenty minutes, or sometimes from ten to fifteen hours, according to the activity of the sample. These crystals are the result of the solution, in the water, of a portion of the cement, and their growth and interlocking on adjoining grains unite the whole into a solid mass, and thus constitute the setting properties of cement. When once the crystals have formed they cannot be again dissolved; hence the worthlessness of a cement which has been remixed after the setting has taken place.

The present **test** of the setting of cements has much practical importance. It is *direct* so far as its limits relate to the time necessary to get the cement in place after mixing, which must not be longer than the time of initial set. It is *indirect* in so far as its limits relate to the probable final strength, elasticity, and hardness of the cement mixture.

One of the apparatus devised for ascertaining the setting time of cement is the

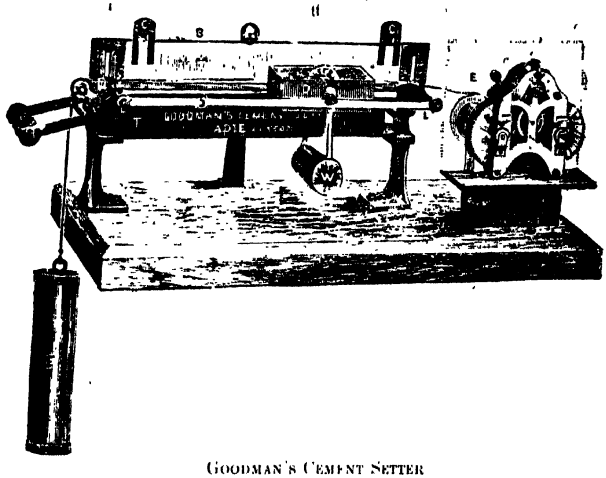
GOODMAN'S CEMENT SETTER,

by which the time is autographically recorded. Although a very ingenious arrangement, this method of testing

cement for setting properties appears to have been little adopted.

An illustration of the device is here given, and the working instructions are as follows :—

Wind up both barrels of the clock. See that all the working parts, especially the compensating levers L L, are well lubricated and are working freely.



GOODMAN'S CEMENT SETTER

Attach a piece of stiff white paper or smooth thin cardboard to the recording board B by means of the clips C C, bring the side bars S to their topmost position and run the carriage D along to make the pencil draw the upper line of the diagram.

Remove the weight W and carriage D, lift out the trough T, and *thoroughly grease* it before putting in the cement with a trowel. Smooth the cement off flush with the top of the trough, and wipe off all superfluous cement.

Replace the trough and the carriage, putting the latter at the clock end of the trough, wind up the string by turning the drum E, hang on the weight F. Oil the disc G all over and replace in the carriage, hang on the weight

W and lower the disc into the cement by lifting the balance weight H; adjust the pencil spring and allow the setter to run undisturbed.

The carriage and disc travel at the rate of one inch per hour, hence the horizontal distance the pencil has travelled before it reaches the upper line of the diagram is the length of time in hours the cement has taken to set, and the slope of the curve shows the rate of setting at each instant.

Full particulars, such as the percentage of water used in gauging, the temperature of the air and the hygrometric state of the atmosphere, should be noted on each diagram.

Another similar apparatus has been introduced on to the market more recently, and is named the

AMSLER-LAFFON CEMENT-SETTING RECORDER.

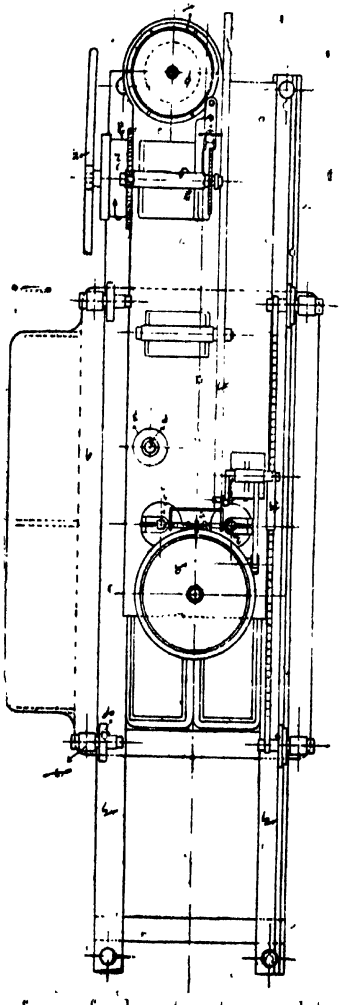
In testing the activity of cements by this apparatus the two moulds are filled with a plastic paste of the sample of cement, and the needles are lowered upon the cement at certain intervals and penetrate more or less into the mortar according to the progress of setting. The depth to which each needle enters into the mortar is recorded on the drum by the pencils.

After each stroke of the needles the drum turns a little, and the table on which the moulds rest advances a step from the right to the left.

The needle holders are lowered and raised by a lever which is set in motion by a spiral spring contained in a box. The motion is set by the clock which lets loose the wing governor and stops it again when the stroke of the lever is completed. The disc with a certain number of notches, is placed on the top of the clock, and performs one turn in an hour. This determines the lapse of time between two consecutive records. By changing the disc different intervals of time may be obtained. The exchange can be carried out during the experiment without disturbing the result, and there are four discs corresponding to 5, 10, 20 and 30 minutes.

When the moulds have travelled from one end to the other, after about fifty strokes of the needles, the carriage

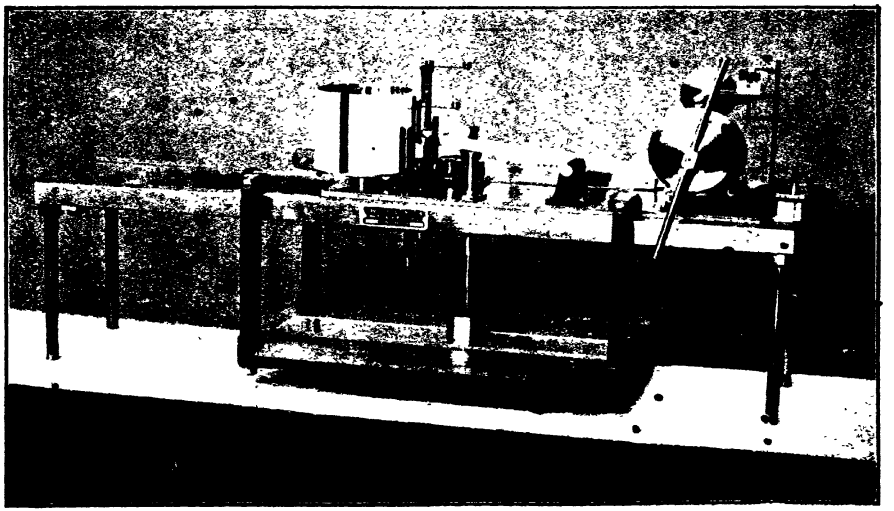
must be pushed back by hand into the initial position and the moulds moved sideways. The diameter of the needles is one millimetre, the weight of each needle-holder is 300 grams. The spring motor is, however, sufficiently powerful to overcome heavier weights, so that the holders may be made heavier if desired.



PLAN OF AMSLER-LAFFON APPARATUS.

The pin V, one centimetre in diameter and 400 grammes in weight, is used for ascertaining the right consistency of the mortar before setting. If the mixture of cement and water is correct the pin ought to stick in the mortar at about one centimetre before reaching the bottom of the mould.

A more common form of testing for setting is that by which the **thumb-nail** is used on a pat of cement, and noting the times of resistance to pressure, thus: a small sample of cement is made up with about 25 per cent. of pure fresh water at normal temperature and formed on



AMSLER-LAFFON CEMENT-SETTING RECORDER.

a piece of glass into a circular pat of about three inches in diameter, with thin edges, by about one quarter inch thick. The initial and final setting times can then be very approximately ascertained by the impressions of the nail at varying intervals, or, more preferably, by a pencil point.

The ascertaining of the setting time of Portland cement, it will have been seen, is still a controversial point, and present methods are still unsatisfactory. Whilst it is without doubt most essential that some definite method of ascertaining and recording the final set and hardening should obtain, it is at least of equal importance to ascertain when the initial set of cement begins, for no user of cement can continue to mix up or work cement after the initial set has commenced without serious injury to the work upon which he is engaged.

If it were wished to improve the setting test it would be necessary not only to measure the time after which the cement has acquired a determined set, such as that defined by the Vicat needle, or by thumb-pressure, but also to measure the **hardness** that it has acquired after a determined time; as, for instance, the result shown by resistance to crushing or **transverse strain** in lbs. per square inch; for, just as it is necessary to know the initial setting time of a cement to save unsatisfactory consequences, so it is equally necessary to know its hardening properties, which quality is of equal, if not of greater importance to the engineer and contractor, who must get through a given amount of work within a certain time. It will be observed that there is something more than the initial and final setting to be ascertained in testing the activity of Portland cement, and that is, the period of quiescence between the final set and the ultimate hardening of the product.

The gift of a strong imagination is not essential in foreseeing the deplorable results that would ensue to engineer, contractor, and cement manufacturer alike where an important concrete construction was in hand, such, for instance, as some heavy re-inforced bridge work, through

this inordinate delay in the **hardening** of a cement, where a product as has been described was employed. In such a case the initial and final setting of the cement could be quite normal, but the hardening of the concrete might be quite another matter, and in the event of centering being removed as might be ordinarily practised, results such as have been outlined are quite within the bounds of possibility.

It may be said that the tensile strength at three and seven days clearly denotes the hardening properties of a cement, but then a cement will often stand a heavy tensile stress whilst still in a green or unmatured state, and this is particularly noticeable with some rotary cements. Some cements of this class, although setting poorly and giving a weak test-pat, may attain a high strength when tested as briquettes, although the final hardening has not taken place. Then also in testing at early dates, say before three days, the ordinary testing machine puts an immediate and heavy strain on the briquette before the test starts, and in the early stages of hardening a record is thus unavailable.

A test is, therefore, required to gauge the hardening of cements, say from the time of final set up to seven days or even longer at short intervals, and, therefore, the apparatus designed for such a test must be one by which the strain can be applied more quickly, more lightly, and at easier stages than is the case with the ordinary tensile testing machine, and still be much more direct and quantitative in its information and results, especially upon the point of the hardening qualities of different cements at varying periods at early dates. A material factor in the setting of cements could then be more carefully studied and the results would be extremely valuable.

With this end in view the author has devised a **transverse** testing machine for cement bars, the working of which is explained in another chapter, and the variable results in the hardening of cements—a point of considerable importance to the constructional engineer and contractor—here show the necessity of such a test, having in view a more careful study of the newer rotary kiln product increasingly employed at the present time.

This test increases the exactness of our knowledge upon the setting of cements, and its results show the hardening properties of cements as well as the setting of the cement under test.

The question arises, however, what setting time should be insisted upon? It is important to have as slow a setting as possible, but limits are put to this by the contrary necessity of having a good strength at the end of not too long a time. Neither the ordinary initial nor final setting tests show this result, and whereas some cements show the initial and final set within reasonable limits, other brands harden at periods varying from some hours to days and even to weeks. The test for transverse strain now advocated is mechanical, and not subject to the will of the operator, and it combines all the tests now outlined that are required in connection with the setting of cements; it is less liable to error in manipulation, and its records are unmistakably efficient in testing both the setting and hardening properties of cements, the latter of which is a knowledge the smallest cement consumer cannot afford to neglect in these days of extensive ferro-concrete construction, where so much depends upon not only the "setting," but the "hardening" of the cement to be employed.

It is rarely that a test of **temperature during the setting** of cement is carried out, but where such a test may be required a method is adopted of applying a small glass thermometer to the block of cement when freshly mixed and placed in, say, the Vicat mould as used for the setting test. Upon the insertion of the thermometer into the plastic cement the temperature is taken and noted, and it will then be observed that the temperature during setting slowly rises until it is distinctly seen that hardening off has commenced in the cement under test, at which point the temperature will be noticed to become stationary. This point of temperature—and before the mercury descends—should be noted, as should also the time of the insertion of the thermometer and the time of the completion of the test.

The results of such tests from a variety of brands at present on the market will probably be as follows, the

length of time in taking the test being indicated and determined by the falling mercury in the thermometer, viz. :—

SLOW SETTING CEMENTS WITH AN INITIAL SET OF ABOUT THREE HOURS (VICAT). INCREASE OF TEMPERATURE DURING SETTING.

Degrees Fahr. Increase During Setting	Length of Test in Minutes	Degrees Fahr. Increase During Setting	Length of Test in Minutes
5	23	4	13
4	8	1	10
3	15	3	14
4	16	3	14
5	23	1	10
2	12	4	20
6	12	4	14
4	25	4	14
3	37	4	9
4	25	4	9
4	17	8	73
6	13	6	84

QUICKER SETTING CEMENTS WITH AN INITIAL SET OF ABOUT SIXTY MINUTES.

Degrees Fahr. Increase During Setting	Length of Test in Minutes	Degrees Fahr. Increase During Setting	Length of Test in Minutes
12	50	7	74
13	36	18	19
11	77	15	64
16	31	13	36
9	21	12	50
11	20		

The present utility of this test is obscure, and its variations cannot yet be gauged or given any theoretical or scientific explanation, nor indeed can any practical information be obtained from the results, for both sound and unsound cements appear somewhat similar in their variations under this test. It is reasonable to assume, however, that the quicker setting cements show

an increased temperature during the setting on account of the exothermic chemical reaction, the thermal unit being practically the same for all brands of Portland cement. The only distinction in temperature is caused by the period during which heat is evolved; for instance, with cement of great initial activity (in other words, in cement of quick-setting properties) the heat is developed in a short period, and consequently shows itself in a rise in temperature, while with more coarsely-ground cement of poorer quality and tensile strength the evolution of the same amount of heat spread over several hours would not be noticeable. The temperature of cement in setting, therefore, is perhaps an index as to the setting times of the cement and not of its hardening qualities.

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Soundness, Contraction and Expansion.

CHAPTER XXI.

THE most useful tests for cement are those which connect themselves definitely with some serious defect to which cements are subject, or with some merit which they should possess; and thus, perhaps, the test for soundness (which should include also the expansion and contraction tests) is of paramount importance. The soundness test can be made with the most simple apparatus—if, indeed, any apparatus is required at all—is easy of manipulation, and its results are obtained immediately. Again, some of the tests which are explained under this heading are comparatively free from personal equation and errors due to local surroundings.

The test for soundness at once obviates many difficulties which arise in the long-time tensile tests for the acceptance or rejection of cements, and the issue of this test is finally decisive.

Numberless methods are in vogue for ascertaining the soundness of cements, and the enquiries more generally classified under this heading are those which have reference to constancy of volume, and to this end the **accelerated or boiling tests** are employed in order to ascertain the exemption of the cement from undue expansion or contraction.

The accelerated tests most generally used are those of putting the cement pat or block in steam and in hot and boiling water. Practically, the steam and boiling water tests may be considered as one, conducted with more or less severity, which increases with the temperature of the water, the steam test being equivalent in severity to a hot water test at about 180° or 190° F. Thus some samples of cement fail in steam and pass in boiling water, although the reverse condition is often found. The steam test, on the other hand, although less severe, has the advantage of being capable of more exact standardisation.

Although, it is true, some rather surprising results are often obtained in experimenting on the soundness of cements, yet it has been shown that a cement that is not quite sound might actually be improved in the steam and boiling tests by the addition of a small amount of lime, and continuing on these lines it has been found in some cases that a cement actually unsound in the boiling test could be made sound by small additions of other brands of

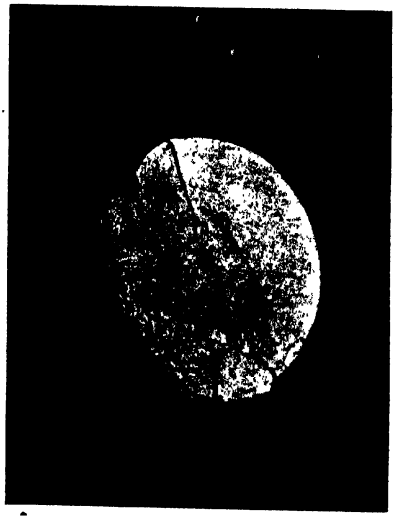


CONTRACTION CRACKS IN CEMENT PATS.

cement, showing that occasionally **a blend** may be more sound than one regularly manufactured product alone.

Contraction cracks in the test pat are often met with but are rarely a sign of bad quality. They are perhaps more generally found to be due to faulty testing rather than to a deterrent quality in the nature of the cement. The contraction crack often seen in the test pat is due to a physical change, and is caused by the lack of humidity of the atmosphere, by the rise and fall of the temperature in the test-room, and draughts of warm or cold air passing

over the pat whilst setting, or again a vibration of the pat whilst the cement is setting will often cause a cracking similar to a contraction crack. A cement which is over-clayed will contract, but contraction cracks are found also if the test pats, when moist, are left in the sun or in a dry atmosphere, or even if the drying takes place too rapidly, and again may be caused by placing the mixed



Expansion Cracks.

cement on a dry, porous plate; an excess of water, too, in the gauging will cause a shrinkage or contraction.

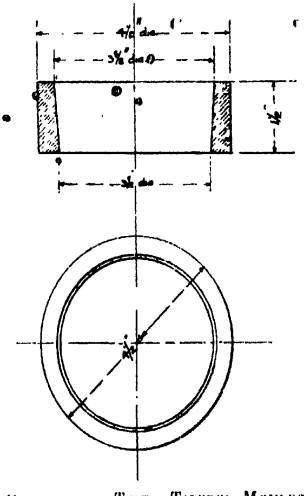
These contraction cracks, as distinguished from those caused by expansion, are irregular lines in the *centre* of the sample pat.

The unsoundness and expansion of cements take many forms, an **expansion** crack, however, will be found to commence upon the *outer edges* of the pat, and run towards the centre. This is by far the more serious defect.

A sure method of testing the contraction of cements is

in the use of a tapered mould, as illustrated. This is filled with neat cement of a plastic consistency and is left to dry off and set hard. If there is any contraction, the cement block will be loose, and will leave the mould.

The expansion of cements is generally caused by what is known as "free" or uncombined lime, and this is intro-



Contraction Test. Tapered Moulds.

duced into the product either by the careless or unscientific mixing or burning of the raw materials used in the manufacture, or by the incorporation of under-burnt clinker from the kilns.

If a cement is poorly manufactured, the aeration or atmospheric slaking will eliminate, to some extent, what free lime may be present, but a properly manufactured cement will withstand the hot or boiling tests as soon as the material is taken from the grinding mills, and any period of aeration other than the time elapsing between the grinding into the manufacturers' stores and the loading of the cement for transport is unnecessary.

Portland cement after having once set must not soften, as sometimes happens, and for some time lose its hardness in order to commence later another hardening process. This defect is always due to the presence of free lime, or uncombined magnesia, the slow slaking of which causes partial disintegration of the already hardened mass. The expansion test with boiling water, however, offers every guarantee against this absence of durability.

Regarding the comparison of soundness tests with the other physical tests of Portland cement, and with the actual constructional experience, we still have the same seemingly hopeless maze of contradictions and irregularities that all cement manufacturers and extensive users are so familiar with.

As to the test for expansion, however, it cannot be stated with any degree of certainty that results which may be obtained by the "hot" test—the now general test for soundness—are identical with those which may be experienced in actual construction work, but there is no doubt that if a cement is in the slightest degree unsound, the hot test will at once detect and emphasise the weakness.

Although, scientifically speaking, there is no **constancy of volume** in Portland cement—since the action during the setting as well as heat and cold, will modify the volume—yet practical results are obtained from the observation of pats of cement as used for the setting tests, and kept for this purpose in air, or immersed in water.

In a practically sound cement the difference in volume is not noticeable without the aid of scientific apparatus, and the value of the test for constancy of volume consists in ascertaining the more common defects in the undue contraction and expansion tendencies of Portland cement.

In the early days of cement manufacture most of the trouble of cement users was due to the expansion of the material through **over-liming** and other faults in the manufacture. This caused a swelling, and ultimate disintegration after the work was in place. An ancient device for the testing of over-limed or expansive cements was to mix a sample of the material with water to about the consistency of treacle, the whole being then poured into an ordinary **bottle** or small glass **test-tube** which is shaken until full to overflowing, and then placed aside in a cool

place. If the cement is *over*-limed, or even if the proper grinding and amalgamation of the raw materials have not been properly carried out by the manufacturer, the cement will expand, and split the glass sooner or later, according to the degree of the imperfection. If, on the other hand, the cement is *under*-limed (or over-clayed), it will contract and become loose in the receptacle.

In these tests care should be taken to keep the bottles or tubes in a cool place or in an even temperature; for, if placed in a warm atmosphere or near a fire, cracking will result through the different rates of expansion of glass and cement even if the cement be perfectly sound.

Another simple test for the observation of the behaviour of cements is to make up two small pats—each about three inches in diameter and one-quarter inch thick in the centre, diminishing to very thin edges. The pats should be gauged and placed upon pieces of glass, and as soon as set—or, say, 24 hours after gauging—one of the pats is immersed in water, and the other left in the atmosphere.

Both pats should be carefully examined at regular intervals over a period of seven days, and the first indication of any disturbance or disintegration will be detected by the appearance of small cracks round the edges, or extending from the centre of the pats. Other alterations of form will follow, such as the lifting up at the edges, or in the centre of the test pat.

The best Portland cement will show no signs of cracking, scaling, crumbling, or warping; nor, indeed, will it suffer any deviation of form whatever under this test.

Any defect in the sample will first appear in the pat under water; but since, in this mild test, it often happens that the evidences of unsoundness are long in appearing, this method has given way to the accelerated or hot-water tests previously mentioned.

The great value of the hot test lies in the short time which elapses before the indications of defects begin to appear, and thus attention is at once directed to weak points in the cement to be further observed or guarded against.

Cements which stand the **accelerated tests,** by steam or boiling water, should be used in preference to others,

and such tests should be constantly applied on the work of concrete construction; for although the hot tests sometime reject suitable material, they will always reject an unsound material by reason of the existence of active expansion.

The decisive test on the Continent for cement soundness is that a pat of neat cement placed on a glass plate shall display when kept under water, no warping or cracking at the edges.

To carry out this test the pat prepared for determining the setting time is, in the case of slow-setting cement, put under water after 2 hours, in no case however before setting has taken place. In quick-setting cement this may take place after a shorter interval. The pats, especially of slow-setting cement, must be protected from draught and sunshine until setting has occurred, which is best secured by storing in a covered box or under moist cloths. The formation of cracks due to **shrinkage**, which as a rule occur in the middle of the pat, and are taken by the inexperienced to be due to expansion, is avoided by these means.

If, when hardening under water, a warping or cracking at the edges appears, this denotes without doubt an expansion of the cement, *i.e.*, in consequence of an increase of volume there takes place a splitting of the cement with gradual loosening of the previously acquired cohesion, which may lead to a total crumbling away of the material.

Evidences of expansion usually show themselves in the pats after three days; but in any case a period of observation extending to 28 days is considered necessary.

In the **United States** the terms of the standard specification in regard to the tests for constancy of volume are as follows:—

Significance.—The object is to develop those qualities which tend to destroy the strength and durability of a cement. As it is highly essential to determine such qualities at once, tests of this character are for the most part made in a very short time, and are known, therefore, as accelerated tests. Failure is revealed by cracking, checking, swelling, or disintegration, or all of these phenomena.

A cement which remains perfectly sound is said to be of constant volume.

Methods.—Tests for **constancy of volume** are divided into two classes: (1) normal tests, or those made in either air or water maintained at about 21° Cent. (70° Fahr.), and (2) accelerated tests, or those made in air, steam, or water at a temperature of 45° Cent. (115° Fahr.) and upwards. The test pieces should be allowed to remain 24 hours in moist air before immersion in water or steam.

For these tests, pats about 7½ cm. (2.95 ins.) in diameter, 1¼ cm. (0.49 in.) thick at the centre, and tapering to a thin edge, should be made, upon a clean glass plate [about 10 cm. (3.94 ins.) square], from cement paste of normal consistency.

Normal Test.—A pat is immersed in water maintained as near 21° Cent. (70° Fahr.) as possible for 28 days, and observed at intervals. A similar pat is maintained in air at ordinary temperature and observed at intervals.

Accelerated Test.—A pat is exposed in any convenient way in an atmosphere of steam, above boiling water, in a loosely closed vessel, for three hours.

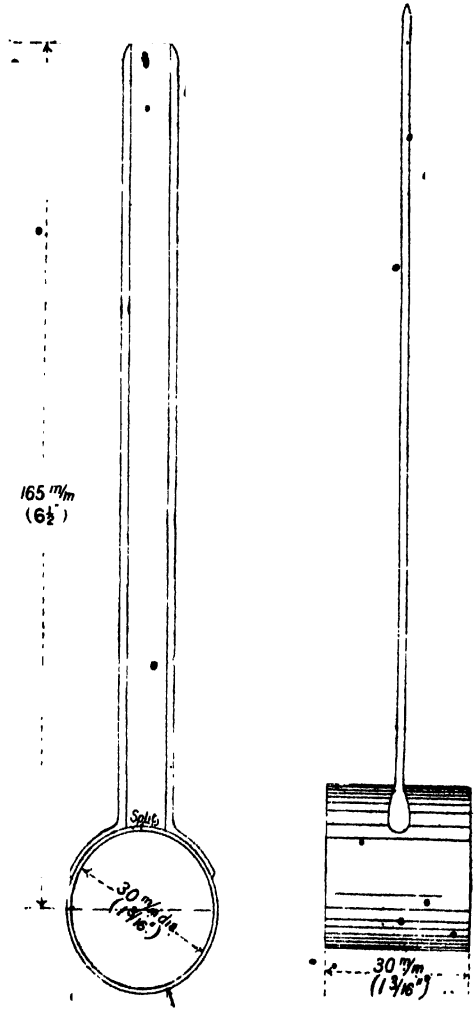
To pass these tests satisfactorily, the pats should remain firm and hard, and show no signs of cracking, distortion, or disintegration.

Should the pat leave the plate, distortion may be best detected with a straight-edge applied to the surface which was in contact with the plate.

In the present state of our knowledge it cannot be said that cement should necessarily be condemned simply for failure to pass the accelerated tests; nor can a cement be considered entirely satisfactory, simply because it has passed these tests.

In the **British Standard Specification** for Portland cement, the soundness test specified is that the cement shall be tested by the Le Chatelier method, and shall in no case show a greater expansion than 10 millimetres after 24 hours' aeration or when this is exceeded 5 millimetres after seven days' aeration.

The apparatus for conducting the Le Chatelier test, shown on the following page, consists of a small split cylinder of spring brass or other suitable metal of 0.5 millimetre (.0187 inch) in thickness, forming a mould 30



LE CHATELIER APPARATUS.

millimetres (1$\frac{3}{16}$ in.) internal diameter and 30 millimetres high. On either side of the split are attached two indicators with pointed ends A A, the distance from these ends to the centre of the cylinder being 165 millimetres (6$\frac{1}{2}$ in.).

In conducting the test, the mould is to be placed upon a small piece of glass and filled with cement gauged in the usual way, care being taken to keep the edges of the mould gently together while this operation is being performed. The mould is then covered with another glass plate, a small weight is to be placed on this, and the mould is then to be immediately placed in water at a temperature of 58° to 61° Fahrenheit, and left there for 24 hours.

The distance separating the indicator points is then to be measured, and the mould placed in cold water, which is to be brought to boiling point in 25 to 30 minutes and kept boiling for six hours. After cooling, the distance between the points is again to be measured; the difference between the two measurements represents the expansion of the cement, which must not exceed the limits laid down in the specification.

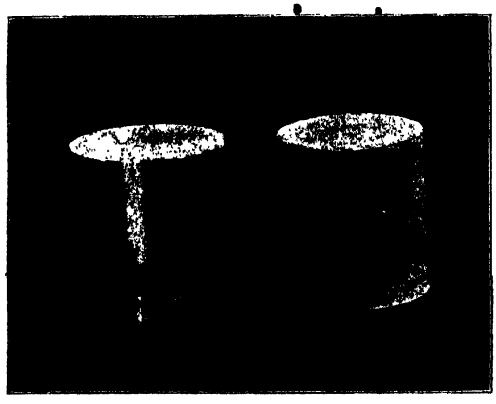
The "Le Chatelier Test."

The test of **expansion** in Portland cements by the Le Chatelier method has been subjected to a considerable amount of criticism on account of its alleged uncertainty, or inaccuracy, or undue severity; and although, in the main, such criticism has been confined to private correspondence, recently published information upon this test gives a more definite pronouncement upon the results of testing various cements for expansion by the Le Chatelier method.

This position has perhaps arisen on account of the generally accepted idea that the results of cement tests by the Le Chatelier method generally show the practical soundness or otherwise of the cement under test; but the actual basis of variance between the advocates and opposers of the Le Chatelier test has, so it is maintained, all along been the question as to whether the limited increase in volume of cements in boiling water generally combines with it the unsoundness or undesirability of a cement in

practice, and not solely in the test itself, which is certainly one for showing expansion pure and simple and not necessarily "unsoundness." So the test as applied to expansion alone will here be dealt with, since many sound cements (in practice) also show a heavy expansion by this test.

It has been maintained, for instance, that numerous Le Chatelier tests of cements made by the rotary process during the last two or three years go to show that the expansion of cements (sometimes very considerable) is not



Le Chatelier Cement Blocks, Showing an Expansion of 12 mm. and 15 mm. respectively after Boiling

always or necessarily due to free or loosely combined lime, but may be due to changes in the fine particles (or clinker grit) under the influence of boiling water, and explanations are given of the theory of these changes. From the results of some hundreds of tests by the Le Chatelier method on cements, principally English-made, but including a few of French manufacture, it has been concluded that cements which have been ground from clinker well and properly burned in the rotary kiln do not improve on aeration, but on the contrary, sometimes become expansive in volume.

It has also been held that the thinner the layer in which the cement is laid out for aeration, and consequently the more it is aerated, the greater will be the expansion by the Le Chatelier test, and suggestions are therefore made by certain advocates to the effect that the British Standard Specification should be modified by providing that cements should be gauged when fresh, or after being spread for 12 hours to 24 hours only. Then, again, the total expansion by the Le Chatelier method in both cold and boiling water, it is maintained, should be measured, the first measurement being taken as soon as the cement has set. The aeration of the cement for 7 days should also be optional.

In following up these conclusions, however, with the testing of other cements, and with a view to checking the results and the theories involved in these contentions, prolonged investigations were instituted by the author, with the result that the findings of **expansion with aeration** cannot be generally confirmed, and it would appear that neither rotary nor other kiln cements expand materially, if at all, upon aeration.

It is now shown, for instance, that the behaviour of fresh and aerated cements in the Le Chatelier test are perfectly clear and unmistakable, and at once indicate the degree of expansion—not necessarily unsoundness—of all cements tested by this method, and many curves have been prepared showing that in both rotary and ordinary kiln cements, while aeration may not show a reduced expansion in a cement by the Le Chatelier method, it certainly does not show an increased expansion from the average results of a considerable number of tests.

In the first place, then, it is interesting to enquire into the effect of the aeration of other than rotary kiln cements as far as expansion is shown by the Le Chatelier tests, and for this purpose a highly expansive cement—not rotary burned—was specially obtained.

The tests (Table I.) appear to show that in a shaft or ordinary kiln cement showing excessive expansion by the Le Chatelier method, the reduction after 28 days' aeration in bulk was from 36 mm. when fresh to 5 mm. upon ageing. After spreading out the same cement the expansion

was reduced from 30 mm. to 4 mm. in the same period, viz. :—

TABLE I.

LE CHATELIER TEST—24 HOURS IN COLD WATER AND 6 HOURS IN BOILING WATER.

	Fresh	3 Days' Aeration	7 Days' Aeration	14 Days' Aeration	21 Days' Aeration	28 Days' Aeration
	mm.	mm.	mm.	mm.	mm.	mm.
Cement in bulk ...	36	33.5	31	20	12	5
Cement aerated 1 in. thick ...	32	30	27	15	7.5	4

Similarly (Table II.) in the case of what may be called a sound rotary kiln cement, the storing in bulk was found to reduce an expansion of 1.75 mm. to one of 0.5 mm. at 21 days, and to show no expansion in 28 days. The rotary kiln cement when spread out reduced in expansion from 1.75 mm. to 1 mm. in 3 days, and no expansion was shown in the tests from 7 days up to 28 days, viz. :—

TABLE II.

	Fresh	3 Days' Aeration	7 Days' Aeration	14 Days' Aeration	21 Days' Aeration	28 Days' Aeration
	mm.	mm.	mm.	mm.	mm.	mm.
Cement in bulk ..	1.75	1.5	1.0	0.75	0.5	Nil
Cement aerated 1 in. thick ...	--	1.0	Nil	Nil	Nil	Nil

To ascertain whether the same results may be expected from a similar treatment of other ordinary kiln and rotary kiln cements, numerous additional tests have been carried out with further well-known brands of cement on the market, and the aeration of these cements gives somewhat the same results by the Le Chatelier test as previously shown.

It is a fact, however, that exceptions have been obtained to the apparently constant diminution of expansion in cements with aeration as indicated in the tests now given, and in one case a rotary kiln cement and an ordinary kiln cement were both found after a reduced expansion with age to revert, after 35 days' aeration, to the original (not increased) expansion as shown by the Le Chatelier test

when fresh; and two days afterwards this expansion was reduced, again showing a continued reduction up to 42 days' aeration.

In considering the results of numerous and carefully conducted tests, such as these, it cannot by any means be admitted that a rotary cement invariably shows more expansion by the Le Chatelier test after a time allowed for aeration than it does when fresh, and it would appear that in both rotary and ordinary kiln cement if aeration may not generally show a reduced expansion in a cement when tested by the Le Chatelier method, it certainly does not show an increased expansion in the considerable number of tests which the author has conducted from time to time.

This being the definite result of these special tests, it serves no useful purpose, other than one of purely academic interest, to enquire into the question as to whether an under-burned or well-burned clinker shows a greater or less expansion by the Le Chatelier test. The general experience in this respect is, however, rather in favour of the reduction of expansion by harder burning.

But to come to the Le Chatelier test results from the division of cements into the varying grades of fineness, such as (*a*) flour, (*b*) clinker grit, and (*c*) sieve residue, we have here three definite degrees of grinding which it is always interesting and instructive to make use of, when examining any cements, by this method for expansion.

The amount of residue which is left on the 180 by 180 mesh sieve in sifting a cement is the standard method of testing for fineness, and it will generally be found that this residue does not exceed 14 per cent. of the quantity of cement tested. From the cement passing through the 180 sieve, the percentage of fine clinker grit in a cement may be collected and checked by resorting to the use of the flourometer, which under a steady pressure of air separates the flour from the fine grit, and the percentage content of the latter in a cement can be accurately ascertained by weighing the fine grit remaining in the flourometer cup after the operation of testing for "flour."

The percentages of "fine grit" and "flour" in a cement necessarily vary according to the degree and process of grinding, but as an illustration of what every-day results may be expected the following quantities of residue, fine

grit and flour were contained in two cements ground by different plants, viz. :—

	Residue 180 × 180 %	Fine Grit %	Flour %	Total %
Ground by tube mills ...	13.4	21.4	65.2	100.0
Ground by other mills ..	13.4	26.3	60.3	100.0

Now, it is a somewhat easy task to ascertain by the Le Chatelier test the identical expansive qualities of each of these species of the genus cement, and after carefully carrying out such a series of tests upon two brands of cement—one rotary and the other not—these results are collated, showing that :—

(1) Two cements testing respectively 2 and 3.5 mm. when fresh are reduced in expansion after each seven days' aeration test up to 35 and 28 days respectively, when the original expansion is again shown, but which at 42 days in both cases is reduced to 1.0 and 1.5 mm.

(2) The expansion in the residue from the 180 mesh sieve with both samples is reduced from 25.25 and 23.75 mm. when fresh, to 21 and 23 mm. after 35 days.

(3) The expansion of the cement passing through the 180 sieve is reduced upon aeration from 1.5 and 2.75 mm. respectively to 3.0 and 1.0 mm. at 35 days.

(4) The expansion of the flour in the rotary cement is reduced from 2.25 mm. when fresh, to 2.0 mm. after 35 days, and in the ordinary cement the flour increases in expansion from 1 mm. when fresh to 2.0 after 35 days' aeration.

(5) The expansion of the "fine grit" also reduces upon aeration from 38.75 and 21.0 mm. when fresh, to 36 and 7 mm. respectively at the end of 35 days.

Thus, when two samples of fresh cement are tested, the relative degree of expansion in the finely-divided component parts is greatest in the coarser residues and least in the finished cement from the rotary kilns. In the ordinary kiln cement, however, the constituent having least expansion appears to be the flour, after which comes the finished cement and finally the grit with the largest expansion.

It seems obvious from these results that the presence

of the flour, the preponderating element in the finished cement, prevents the expansion of the grit to a very large extent in the combined sample as compared with the expansion measured when the grit alone is tested. If this were not so the expansion due to the grit alone in a finished sample of cement would be much greater than is actually observed.

The following table shows the results of the tests referred to viz. :—

LE CHATELIER TEST.

EXPANSION IN BOILING WATER FOR SHOWING AERATION RESULTS UPON COARSE AND FINE PARTICLES.

	Period of Aeration					
	Fresh	7 Days	14 Days	21 Days	28 Days	35 Days
	mm.	mm.	mm.	mm.	mm.	mm.
Rotary kiln cement ...	2.0	1.5	1.5	1.5	1.25	2.0
Residue on 180 × 180 ...	25.25	22.5	23.5	24.0	21.5	24.0
Cement through 180 × 180 ...	4.5	3.75	2.0	1.75	3.0	3.0
Grit from flourometer ...	38.75	43.5	41.0	39.0	37.5	36.0
Flour from flourometer ...	2.25	1.5	1.25	2.5	1.75	2.0
Ordinary kiln cement ...	3.5	2.0	2.5	2.25	3.5	3.5
Residue on 180 × 180 ...	23.75	20.25	24.25	21.5	27.0	23.0
Cement through 180 × 180 ...	2.75	2.25	1.0	2.5	1.0	1.0
Grit from flourometer ..	21.0	20.0	18.5	12.5	9.0	7.0
Flour from flourometer ..	1.0	1.5	1.0	1.75	2.75	2.0

The individual results from the Le Chatelier test of all the component parts of ordinary Portland cement are interesting, therefore, only in their waverings and non-calculable results, but this is inevitable in all tests into which an equal amount of the personal element is introduced in testing, and to show, in addition, what further peculiar results it is possible to obtain by the Le Chatelier test for expansion through other methods by other operators and from other cements, a few moments' consideration of the results of many tests in this direction will give the student some idea of the futility of an attempt at appraising the test upon any ordinary theoretical or even practical conclusions, whether based on a chemical or physical evaluation.

The inferences to be drawn from such tests therefore are that Rotary and other cements do not increase in

expansion with age, for with few exceptions only an ultimately reduced expansion by aeration is generally shown by the Le Chatelier test.

Again, considerable variation is often found in the results of tests by the Le Chatelier method, as carried out by the same operator on the same cement, as will be seen by the following schedules of the expansion in millimetres upon three samples of cement made by different processes, viz. :—

(*a*) Rotary kiln cement. (*b*) Shaft kiln cement.
(*c*) Chamber kiln cement.

Each sample of cement was divided into five sections and each sectional sample was made up and tested at the self-same time, but at varying dates of aeration. The expansions in cold and boiling water are shown and are totalled together, giving the aggregate expansion in the samples.

Le Chatelier Expansion Tests.

Sample of Cement divided into five parts and aerated, tests being made every three days.

SAMPLE A.—ROTARY KILN CEMENT

Days of Aeration	Sample No. 1			No. 2			No. 3			No. 4			No. 5		
	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total
As Ground	3.0	3.0	6.0	3.0	2.0	5.0	3.0	2.5	5.5	4.0	4.5	8.5	3.0	5.0	8.0
3 days	5.0	1.5	6.5	3.0	2.0	5.0	4.0	2.0	6.0	6.0	2.0	8.0	5.5	1.5	7.0
6 ,,	4.5	2.5	7.0	3.5	2.5	6.0	3.5	2.0	5.5	5.0	3.0	8.0	5.0	2.0	7.0
9 ,,	5.0	2.5	7.5	2.5	2.5	5.0	4.5	1.5	6.0	4.5	2.0	6.5	6.0	2.0	8.0
12 ,,	7.0	2.0	9.0	5.5	1.5	7.0	4.5	2.0	6.5	6.0	2.0	8.0	5.0	.5	5.5
15 ,,	5.0	1.5	6.5	3.5	1.5	5.0	2.5	3.5	6.0	3.0	2.0	5.0	5.0	2.5	7.5
18 ,,	5.0	2.0	7.0	4.0	1.0	5.0	4.0	1.0	5.0	4.0	1.0	5.0	2.0	1.0	3.0
21 ,,	4.5	1.5	6.0	6.0	3.0	9.0	4.5	2.0	6.5	2.0	2.0	4.0	5.0	2.0	7.0
24 ,,	—	1.5	1.5	—	1.5	1.5	—	1.0	1.0	4.0	2.0	6.0	5.0	1.0	6.0

SAMPLE B.—SHAFT KILN CEMENT.

Days of Aeration	Sample No. 1			No. 2			No. 3			No. 4			No. 5		
	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total
As Ground	4.0	5.0	9.0	4.5	3.0	7.5	2.5	5.0	7.5	4.5	3.5	8.0	4.0	4.0	8.0
3 days	1.5	4.5	6.0	2.5	7.0	9.5	1.0	5.5	6.5	3.0	8.0	11.0	4.0	8.5	12.5
6 "	1.0	4.0	5.0	.5	3.0	3.5	1.0	5.0	6.0	1.5	3.5	5.0	.5	5.0	5.5
9 "	2.0	4.5	6.5	2.0	1.5	3.5	1.0	3.5	4.5	2.0	5.0	7.0	3.0	4.0	7.0
12 "	2.5	3.0	5.5	3.0	3.5	6.5	2.0	3.5	5.5	3.5	4.5	8.0	2.5	3.0	5.5
15 "	3.0	2.5	5.5	3.0	3.0	6.0	3.5	3.0	6.5	3.5	2.0	5.5	3.5	2.5	6.0
18 "	3.5	2.5	6.0	3.0	2.5	5.5	3.5	2.0	5.5	1.0	2.0	3.0	2.5	2.0	4.5
21 "	3.0	2.0	5.0	1.5	2.5	4.0	3.5	2.0	5.5	3.5	2.0	5.5	3.0	2.5	5.5
24 "	—	2.0	2.0	—	1.5	1.5	—	1.0	1.0	.5	1.5	2.0	—	2.5	2.5

SAMPLE C.—CHAMBER KILN CEMENT.

Days of Aeration	Sample No. 1			No. 2			No. 3			No. 4			No. 5		
	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total	Water	Boiling	Total
As Ground	2.0	2.5	4.5	4.0	2.0	6.0	3.5	3.5	7.0	6.0	3.0	9.0	5.5	2.5	8.0
3 days	7.0	3.0	10.0	5.5	1.5	7.0	7.5	2.5	10.0	2.0	1.5	3.5	1.5	2.0	3.5
6 "	4.5	2.0	6.5	4.0	2.5	6.5	8.0	1.5	9.5	8.0	2.0	10.0	6.5	2.0	8.5
9 "	.5	3.0	3.5	3.0	2.5	5.5	.5	2.0	2.5	—	2.0	2.0	.5	2.5	3.0
12 "	2.5	2.0	4.5	1.0	2.0	3.0	.5	3.5	4.0	1.5	3.5	5.0	1.0	2.5	3.5
15 "	2.0	2.5	4.5	2.5	2.0	4.5	2.0	1.5	3.5	4.0	1.5	5.5	5.0	1.5	6.5
18 "	—	.5	.5	1.0	1.0	2.0	-	2.0	2.0	.10	4.0	4.10	.5	1.0	3.5
21 "	1.0	2.5	3.5	.5	2.5	3.0	1.0	2.5	3.5	- -	3.0	3.0	1.5	2.0	3.5
24 "	.5	2.0	2.5	1.0	1.5	2.5	1.0	2.0	3.0	.5	1.5	2.0	—	1.5	1.5

It will, moreover, be found from the numerous investigations which have been published by experimenters from time to time in connection with the Le Chatelier test that they clearly show the impossibility of fairly gauging either the minute variations of this test upon ordinary samples of cement, or its discrepancies or idiosyncrasies, because each and every experiment is directly governed, in the case of each sample of cement tested, by, among other points :—

(*a*) The nature of the raw materials used in the manufacture of the cement.

(*b*) The process and plant used in the reduction and mixing of the raw materials from which the cement has been produced.

(*c*) The chemical evaluation of the raw material mixture.

(*d*) The method of burning the mixture, and the plant employed, and the fuel consumed.

(*e*) The degree of final grinding of the clinker and the plant employed.

(*f*) The period of storing and aeration of the cement.

(*g*) The personal equation in carrying out the test, and the percentage of water employed, temperatures, &c.

All these points, with others, are of importance in calculating the value and results of the Le Chatelier method of testing the expansion of cements, but there is no more exception to be taken to this position than there can be in, for instance, testing the tensile strength and setting of Portland cements: and although such tests are not sensitive in the same degree to these numerous conditions, yet the tensile strength test at least is one equally affected by similar conditions in the mean results.

As far as can be seen, therefore, there can be no disputing the value of the Le Chatelier test in ascertaining the expansion of cements, but whether the **practical soundness** of a cement is to be definitely gauged by these expansion results or not is quite a debatable point and this, doubtless, is more the bone of contention—although not so clearly put forward by its advocates—than is the futility of the test itself.

It has been urged, on the one hand, that perfectly sound and satisfactory cements, which will act heroically in concrete work, and will well withstand the test of age, may sometimes expand more than the 5 mm. allowed as the limit by the British Standard Specification; and, on the other, that many cements, which will show little expansion by the Le Chatelier test, are so unsound as to completely disintegrate in boiling water. Practice and experience show that this is, in the main, true, and may be regarded as justifying the contention that this test is purely an expansion, and not a soundness test.

The only practical difference in position, however, between the stringency of the Le Chatelier test and other cement tests is that the five millimetres expansion allowed by the British Standard Specification is a low limit compared to what has been found to be the expansion by the same test in many well-known and long-tried cements which have been used in considerable quantities with sound and satisfactory results in years gone by.

But the modern demand for a limited expansion in cements, it is true, may eliminate a product made by old methods and old processes, and if this position is altered by the immediate adoption of scientific methods and efficient plant, then there is no difficulty whatever in producing a material which will come within the limits of expansion by the Le Chatelier test that are allowed by the terms of the British Standard Specification.

In preparing a cement to pass this test, therefore, there would be no more effort required on the part of the manufacturer than in preparing one to pass the tensile strength test, providing the British Standard Specification was as stringent in its demands of the qualities of the latter as it is in the former, and, indeed, if the tensile strains of cements were raised to modern conditions, we should see the 3 to 1 sand test results well-nigh where the present neat tests stand. This position would be more compatible with the present-day Le Chatelier test requirements, and then the theorising as to the causes and effects in respect of variations by both tests—and even with other tests—could all be dealt with in the one category. They are all more or less governed by the varying conditions and

materials employed in the process of manufacture and by the methods employed in the testing.

It is of as little use to try and assign one common cause to the vagaries of this test as it is to propound an exact theory upon the cause of the ever-varying tensile strength tests or of the setting times of other tests of the same cements, as shown by different operators. All are due to innumerable conditions and circumstances, and the results obtained from testing different brands of cement, as shown by the Le Chatelier tests, as well as by other cement tests, are legion, as must be their causes.

It may be, and no doubt is quite true that in restricting the expansion of cements as shown by the Le Chatelier test to a low limit, many good and long-tried cements will be debarred from entering the field of "expansionless" cements, but—contrary to the statements which have been made from time to time—it is invariably the rotary made cements that can the more readily pass this test, whether freshly ground or aerated.

It cannot be disputed that this method puts the testing of the expansion of cements upon a distinct quantitative basis—an eminently desirable step—certainly at the time of the test, if at no other period, although no such certain statement can be made in regard to any test showing the soundness of cements. It is a test easily manipulated, with results readily calculated, clearly comparable, and at once declaring the cements most free from expansion, even if not the soundest, and so long as the consumer desires to use one product possessing a less expansion tendency than another, with other qualities equal, there is a distinct advantage to be obtained from its use.

All will be willing to agree that absence of expansion is the most essential quality for a cement to possess, and that the ordinary rule-of-thumb tests for soundness as adopted by other specifications are the most inexact and the most dependent of all the routine tests upon personal judgment.

Such a position as regards expansion the Le Chatelier test clearly avoids, and, finally, for ensuring absence of expansion alone, this test, be it said, has done more for the improvement in the quality of cements generally than any other requirement of the British Standard or other cement specification.

The "Cold Plunge" Test.

The **soundness** tests in some Portland cement specifications include the requirement that the cement shall pass the **cold plunge or immediate immersion test.** Such a clause as the following is, therefore, included in some cement specifications, viz. :—

"The cement shall be thoroughly sound and well cooled. Pats on glass made therefrom must show no sign of contraction, expansion, or disintegration whether (1) after being kept in a humid chamber or in water for twenty-four hours, they are placed in cold water, which is then brought rapidly to boiling point, and kept thereat for twelve hours, or (2) they are **plunged into cold water** shortly after being gauged and kept there for any length of time, or (3) they are kept entirely in the air. The boiled pats must not show any tendency to hold water, but must dry immediately on being taken out of the hot water. The 'plunge' pats in addition to not being cracked, warped, &c., must have a firm and permanent adherence to the glass."

"Unsoundness" in the cold plunge test is sometimes looked for in the cracking of the surface of a pat of cement which has been placed under water immediately after gauging, but more particularly in the distortion of the same, the pat at times leaving the glass at the edges. This is a test which many otherwise perfectly sound and expansionless cements often fail to pass, and the scientific reason of so many cements showing a surface cracking under this test is difficult to explain until more exact experimenting has been carried out with a test which comes into demand only in rare instances.

The point to be cleared up before definitely stating the value of the test is chiefly the one of the action of water upon the scum or surface of cement as it is setting, and the cause of this surface cracking in the pats will probably be found from such an investigation to centre itself often, if not entirely, with slow-setting cements, on some mechanical action depending upon the operator or the methods of carrying out the tests.

It cannot, however, be definitely said whether the test shows a defect in the cement or not, but it is certain that

surface cracking is more often caused by the manipulation of the test rather than by any action of the cement under water. Indeed, from an experience of the new vagaries brought into cement testing by this test the defects shown by it up to the present all appear to centre themselves in conditions quite outside soundness altogether.

It is generally assumed that cement containing **gypsum** will fail to pass the cold plunge test, but this is quite a mistake. No doubt such an explanation has been put forward from time to time by those who are anxious to explain away through some reason or another these idiosyncrasies in the testing of cements, but it is far better to grasp the facts and admit the necessity for an enquiry than to base an explanation upon improbable circumstances. Many cements containing as much as 3 per cent. of gypsum will pass the cold plunge test, while again many cements which do not contain gypsum or any other addition to the clinker, whether rotary made or otherwise, will show "skin" cracking and will lift off the glass. It is true that many cements containing gypsum are particularly liable to fail in this test, due perhaps to their slow-setting nature, and also because of the light, fine particles of gypsum and cement being brought to the surface by the trowelling and shaking of the pat whilst in preparation, but from many experiments it seems more reasonable to conclude that the treatment of the pat has more to do with the cracking under the cold plunge test than has the presence of gypsum, although this does not apply to the lifting of the pat at the edges.

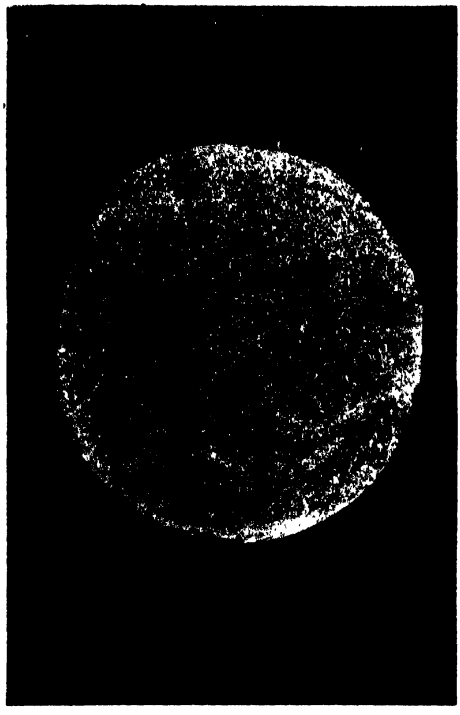
It has also been observed that the temperature of the water into which the cement pat has been plunged is sometimes the cause of the surface cracking by this test. For instance, a number of pats immersed in water with a temperature of 66° F. will be found to be sound by the cold plunge test, while the same cement made into pats will lift and crack on the surface if put into water with a temperature of 45° to 50° F.; and again, it has been noticed that in the case of two pats of the same cement prepared in exactly the same way, if one is put under fresh water and the other put under the older and alkaline water contained in the usual briquette tray, the latter pat will remain sound, while the former is unsound.

Further, the soundness or unsoundness of a pat under this test is controlled to a large extent by the time elapsing between the finishing of the preparation of the pat, and the time at which it is plunged under water. The same cement made into two pats will act as follows under this test :—

One pat put immediately under water as gauged, will show a surface cracking, and the other pat if allowed, before immersion, to get a dull surface after some 15 to 20 minutes in the atmosphere, a condition showing that the initial set is likely to take place within the next 15 minutes, then this pat, when immersed in water, is perfectly sound.

The pats in the cold plunge test are also affected by the vibration of the water in the tank in which they are immersed. For instance, if the tank of water remains perfectly still, and the water is not interfered with, a sound pat will often result, whilst with another pat of the same cement, if placed in a tank where briquettes or other pats are being constantly taken from or put into the water whilst the cold plunge pat is setting, then disintegration of the surface results. Sometimes, again, if a prepared sample of cement is made up into two pats, and one of them be shaken down and the surface trowelled, this one will crack in the cold plunge test; while the other sample of the same cement made into a pat by merely cutting into shape with a trowel, and no trowelling on the face, will, under the cold-plunge test, be perfectly sound. This result of **trowelling** is seen in a more aggravated form in some cements either as briquettes or **pats.** In such cases it is noticed that the " skin " or " scum " on the surface will separate, if there has been much trowelling, and if there has been no trowelling the surface will remain perfectly sound. This defect again—clearly distinguishable from the cold plunge test crack—very commonly occurs where excess of gypsum has been added to the cement, and if a scientific explanation were wanted, the defect might be attributed to the action of sulphur compounds which exist in greater quantity in the skin than in the body of the cement block or pat. This the author has demonstrated by direct experiment with successful small additions of gypsum to different cements, when it was

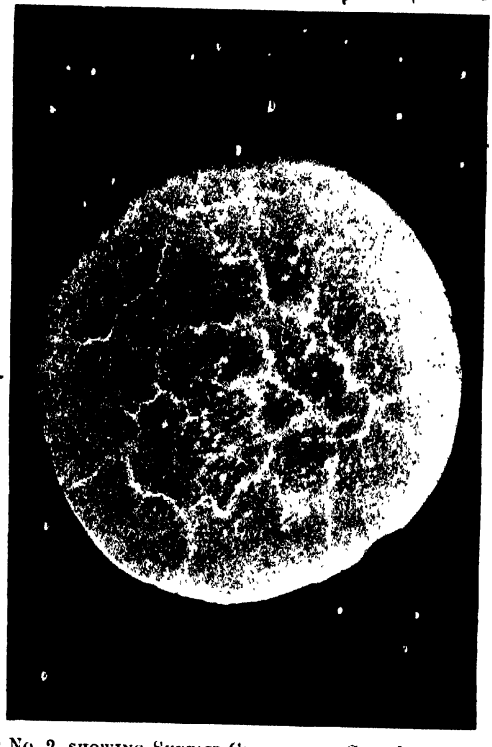
found that ultimately in the majority of cases a time was reached when the skin of a shaken or trowelled pat or briquette would part from the body when placed in water after setting.



PAT NO. 1, SHOWING CRACKING BY OVERTROWELLING IN COLD PLUNGE TEST.

In observing the cold plunge pat test, therefore, some important regard should certainly be paid to the results from the trowelling of the surface of the cement pats, for a distinction can always be made between a cracking caused by overtrowelling and cracking caused in other

ways. To enlarge upon this point, two photographs are here given of a pat No. 1 where the uplifting skin is shown to be much denser than in pat No. 2, which has not been



PAT NO. 2, SHOWING SURFACE CRACKING IN COLD PLUNGE TEST.

trowelled, and the latter shows what might be called a complete cold plunge test cracking. At the same time another pat of the same cement, as employed in these two pats was perfectly sound under the cold plunge test when placed in the usual laboratory briquette tank, the

other two pats being placed in a separate tank of fresh water by themselves. The cement from which these three pats were made contained gypsum up to 2 per cent.

It will sometimes be seen that with a freshly-made, well-trowelled pat the skin will be denser, and water will work its way through this skin whilst the cement is setting and cause it to expand slightly or lift in so doing, and thus separate from the body of the pat in which the expansion caused by infiltering water would be much smaller. From other observations made in this direction it appears that the difference between the cracks observed on untrowelled pats under the cold plunge test, and those on pats which have been overtrowelled, is that in the former a somewhat general but slight disintegration takes place, resulting in fine cracks on the surface of the pats, and the cement is friable and does not set quite hard, but with the overtrowelled pats a thin skin is seen on the top of the pat, and this alone disintegrates whilst the pat itself remains sound and hard.

In summarising the author's experience of this test, therefore, it would appear that as a broad general rule, only slow-setting cements are affected by the cold plunge test, and this without the quality of the cement being called into account at all. It is clear, however, that under this test defects might be separately or collectively occasioned by :—

(*a*) The result of the fineness of grinding of the raw material used in the manufacture, and either a high or low lime content.

(*b*) The degree of burning of the clinker.

(*c*) The percentage of gypsum which is added to control the setting of the cement.

(*d*) The overtrowelling of the test pat in preparation.

(*e*) The difference between the temperature of the cement when tested, and of the water when the pat is "plunged."

(*f*) The period of aeration and temperature of the cement after grinding.

(*g*) The method and treatment of the pats before and after "plunging."

An examination of the causes of surface cracking in the cold plunge tests shows that from the results of a considerable number of tests; the cause of this cracking may be accounted for in one or other of the defects already noticed, or by a combination of the same. For instance :—

(*a*) If the raw material during the process of mixing is not ground sufficiently fine in its preparation for passing through the kiln, the result is that some particles are not properly burned, as the raw material has not been finely enough divided to form an intimate mixture required to obtain complete chemical combination in the kiln. This results in a quantity of the cement being under-burned where the lime is only partially combined. Such a cement on being subjected to the cold plunge test may cause an expansion which might crack or destroy the shape of the test pat.

(*b*) This to some extent really accompanies the results of the fineness of grinding of the raw material already mentioned, when the clinker is not properly and regularly burned. With raw materials which may be extremely finely ground, but low in lime, there is often an over-burning of the clinker from this cause, and the cement resulting therefrom shows a surface cracking by the cold plunge test. Cracks in such a case do not extend deep into the pat, but the skin can easily be peeled off.

(*c*) In the case of the result of adding gypsum to the clinker when grinding, an unsoundness is sometimes observed in the pats under this test, and which in most cases is shown by the lifting of the edges of the pat off the glass, that is, the pats become distorted in shape, and in some cases gradually leave the glass altogether. The cracking of the surface is only sometimes found, but the usual sign of excessive gypsum adding as shown by the cold plunge test is the leaving of the pat from the glass in its entirety.

(*d*) A common cause of surface cracking in testing cement by the cold plunge test is seen with a quick or medium setting cement. In making a pat and preparing the fine feather edges of the same, the repeated rubbing

of the trowel to smooth off the surface gradually disturbs the set, and practically "kills" this surface of the cement pat. The pat on being "plunged" is some time in setting, and when setting commences small cracks are observed, which ultimately spread all over the surface of the pat or where the greatest application of the trowel has taken place. The skin then splits away from the body of the pat, and in some instances is much thicker than that in the case of the usual cold plunge pat cracking.

(c) The difference of temperature in the cement tested and in the water used, and in the air during the period of cement aeration after grinding, also considerably affects the results of cement pats immersed in accordance with the cold plunge test. In warm weather, for instance, and particularly when the cement is taken warm from the grinding mills, this is often the cause of unsoundness.

A test pat under the cold plunge test is made without special regard to standard temperature either in the cement or the water used in the test, and on these special points great care requires to be taken. Cement, for instance, laid out to cool in the atmosphere remains at the temperature of the atmosphere, and on being mixed this cement may be several degrees higher than the temperature of the water, which is generally cooler than the atmosphere. A pat of cement under these circumstances on being plunged into cold water shows surface cracking after a very short time, and in this respect it has been noticed that where a cement was perfectly sound under the cold plunge test when mixed early in the morning and immediately "plunged," yet when the same cement was mixed in the heat of mid-day and then "plunged," cracks developed over the surface of the pat.

The method of making the pats and the treatment of the plunge pats after making are also a common source of surface cracking by this test. When the cement is mixed with water a portion may receive less water, and consequently set more quickly than the remainder of the cement used in the pat.

Again, in the case of the cement mixed with a minimum of water, the paste is found to be stiffer than the other cement mixed with an excess of water and not so easily smoothed off or shaken down. When such a pat is

"plunged" cracks are often noticed upon the thick part of the pat, but are not existent in the other portion.

The test is also affected by the treatment of the pat when put under water. After the pat has been under water for a time, it is often taken out of the bath to see what is taking place. This repeated motion of the pat and water seems to set up some tension in the surface skin, and so cause it to crack. This is most frequently noticed when any special samples are being treated in the bath at the same time as the cold plunge test; for instance, pats that have been taken out and examined have been observed to be surface cracked, and the same cement in a pat made in exactly the same way which has not been disturbed during its setting under water has been perfectly sound in every way. Similarly, several pats under water have been disturbed by the movement of the water due to taking briquettes from the baths in which the pats were immersed, and in many such cases the pats have developed surface cracks; but fresh pats of the same cement which have been made and placed in the same tank and water alongside those previously cracked, have, where the water has not been disturbed, been perfectly sound in every way.

Then again, there is a certain personal element graduated from experience with this test which can make the cold plunge test either satisfactory or otherwise in the same cement without any obvious difference in the method of carrying out the test. The pats made, for instance, by inexperienced persons have been noticed to be unsound, and the same cement when employed by one who is experienced with the cold plunge test may be perfectly sound in every way. In some cases also some pats when placed in water immediately after gauging remain perfectly sound, whereas the pats of the same cement if kept until the initial set has taken place show decided cracking on the surface and lifting.

These are some of the points which show a hazardous experience of the cold plunge test when such a test is regularly carried out, and the results go to prove that quite a number of conditions have effective influence on the results of the tests.

In all these cases the Portland cements which have been

tested have readily withstood all the standard and other recognised tests for soundness, strength, &c., including the 5 mm. limit of expansion by the Le Chatelier method.

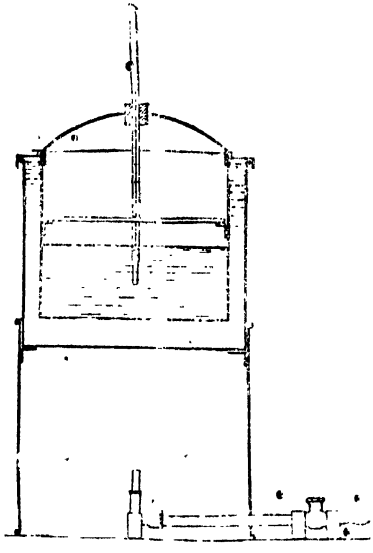
It must, therefore, be held that because a pat cracks and flakes on the surface under this test, it does not by any means follow that this is due to the unsoundness of the cement. The value and results of the test as far as experience in practice is concerned, are equally unimportant, especially when there is a certain doubt as to whether the handling of the cement or the cement itself, apart from the possibility of the defect being calculable or not, is the cause of the peculiarity which is the extent of the result of the surface cracking of pats of cement by the cold plunge tests.

It may be said that the explanation of the behaviour of cements by these tests is caused entirely by the surface contents of the pat differing in composition and constitution from the bulk contents, an experience not found in the practical uses of cement, and whether a cement contains gypsum or not has little bearing upon the results by this test. The remarkable point in regard to the insistence of this cold plunge test by some enthusiasts is the opinion freely expressed that faulty results by the cold plunge pat test essentially show a faulty cement, yet when the test is carried out with a small amount of sand or other admixture, the defect shown in the neat cement cannot occur. The same enthusiasts repudiate neat tests for tensile strength, which are hopelessly condemned as not representing the true qualities of the product and are not according to experience in practice, yet in the cold plunge test the neat cement results are held to clearly show the quality of the product under investigation. Such common inconsistencies in the testing of cements cannot be better illustrated and deprecated.

A well-known test for the soundness of cement is that called the **Faija test.** It consists in subjecting a freshly-gauged pat of cement to a moist heat of 100 deg. Fahr. for six or seven hours, or until thoroughly set, and then immersing it in warm water at a temperature of 115 to 120 deg. Fahr. for the remainder of 24 hours.

This accelerated test is based on the principle that moist heat accelerates the setting of cement, and that, if

judiciously applied, the age of several days may be artificially given to a cement in a few hours. A sound cement acquires great hardness in a short time when treated in this way, but an unsound one, or one that would under ordinary conditions "blow" when used in work, is caused to develop this latter characteristic in a few hours; and hence, by the use of this test, a definite opinion may be



Faija's Hot Water Apparatus.

formed as to whether or not a cement is a safe one to use, independently, of course, of its tensile strength, which may or may not be equal to that required.

The apparatus consists of a covered vessel, in which water is maintained at an even temperature of from 115 deg. to 120 deg. Fahr.; the space above the water is, therefore, filled with the vapour arising therefrom, and is at a temperature of about 100 deg. Immediately the pat is gauged, it should be placed on the rack in the upper part

of the vessel, and in five or six hours it may be placed in the warm water and left therein, for nineteen or twenty hours. If at the end of that period the pat is still fast to the glass, or shows no sign of blowing, the cement may be considered perfectly sound; should, however, any signs of blowing appear, the cement is laid out in a thin layer for a day or two, and a second pat made and treated in the same manner, as the blowing tendency may only be due to the extreme newness of the cement.

Another, and perhaps readier, test for soundness is as follows:—

Prepare sample pats of cement in the ordinary way, and after 24 hours, place them in a receptacle of cold water, which should then be heated gradually to boiling point: allow to boil for two to four hours. If the pats are sound the cement will be found thoroughly satisfactory in the constructional work in so far as constancy of volume is concerned.

Tensile Strength Tests.

CHAPTER XXII.

THE value of a cement depends upon its power of setting into a rigid mass soon after it is gauged, and on its power of attaining a considerable strength in course of time. The testing of the strength of Portland cement may be performed by compressive and by tensile tests, the latter including the transverse test made by breaking a beam of the cement.

In actual work the strain to which cement is subjected is not necessarily a purely tensile or purely compressive strain owing to unequal bearings and loadings, but is more often a transverse strain. The transverse and compressive tests are, however, rarely made, the most usual test for cement, largely for the sake of convenience, being that of its tensile strength.

The compressive stress for testing Portland cement can be readily applied by apparatus designed to this end; but the test serves little purpose since the compressive strength of neat cement is found to be in a fairly constant ratio of about 10 to 1 as compared with the tensile strength, the test for which may be applied more easily and by simpler and more familiar apparatus.

There is, however, likely to be a much greater demand for results of compressive tests with sand and cement, owing to the increased uses of reinforced concrete and to the variable results given by different cements when tested with sand in compression.

At the present time the section of a cement report which receives the most attention is that dealing with the tensile strength. This, perhaps, is not surprising, when we remember that this test, although possibly the most illogical and erratic, is the most convenient.

The object of the tensile (or stretching) test is to determine the greatest stress per square inch which, under given conditions, the cement can be made to stand without rupture. If the conditions have been carefully observed,

and yet several discrepant results are obtained, the highest *may* be right, but the others are certainly wrong. No averaging should be done, although this is a practice regularly resorted to.

In respect of the uncertainties due to the personal characteristics of the tester and to the influence of local conditions, this test offers greater scope for error than any of the others considered.

The most scrupulous care must be observed in the manipulation, and the tester should possess natural aptitude for such work.

There is, fortunately, now a standard method of testing cements in this country, but engineers frequently draw up their own specifications for testing. The average tensile test of neat cement as marketed to-day is from 600 to 700 lbs. per square inch in seven days, and 800 to 850 lbs. in twenty-eight days, these being the two periods most generally adopted for testing, and, on the whole, the most convenient. A tensile strength above the average mentioned need not, however, excite suspicion as to the soundness of the product, as is often the case, for a carefully made cement will often sustain very high strains before fracture; and if such a cement withstands the far stricter accelerated tests for deciding as to the safety of the material, a high strength may certainly be looked for as one of its decided qualities. It is, however, sometimes the case that a high tensile strength may be caused by over-liming the raw material, but since a number of tests are in vogue for indisputably discovering such a weakness in a cement, one can by these means readily confirm the records of the tensile test and prove the soundness of the material.

With the now increasing demand for a finely ground cement, it is to be expected, and in fact it should be urged, that in place of **neat tests** an admixture of sand should be employed in estimating the value of the sample. In the neat tensile test the full value of the cement as a concreting material or as a cementing power never comes into play; and, granted that a finer cement is a more valuable product, a coarse sample will, in a neat test, give results as to tensile strength equal or superior to those obtained by a fine cement. But, on the other hand, the difference

between the constructive values of a coarse and a fine cement will be most noticeable in a test for tensile strength, carried out with a mixture of sand and cement in the proportion of 3 to 1.

The value of sand tests in place of neat tensile tests is more appreciated day by day, and this hardly requires an explanation since cement is rarely, if ever, used in a neat state, except by consumers who have a wrong impression of its qualities. In a sand test the more practical concreting qualities of the product are ascertained and its value as a constructive material is assured. Many cement specialists have consequently ceased to consider the neat tensile tests of importance or of any practical value, and, therefore, on the former count the test of neat briquettes is deprecated.

The experience of those who have spent a considerable part of their life-time in testing cements seems to show that every brand of cement has its own distinct peculiarity which requires to be investigated and understood before it can be stated that any given result by the tensile strength test is a true and sufficient ascertaining of the value of the identical sample under test.

An instance of this has been known to occur where a sample of cement made into a semi-dry briquette will break at some 800 lbs. per square inch, but if made plastic the same cement will break at about 450 lbs. Another sample prepared semi-dry for the neat tensile test will break at about 500 lbs. per square inch, but if made in a condition equal to plastic the same cement will break at some 550 lbs., and yet the results obtained from the sand tests of these two cements have been almost identical.

In the opinion of many who are authorised by experience to give a reliable statement upon the neat tensile strength test, it is held that the extraordinary susceptibility of neat cement renders it impossible to expect any reliable results from the usual haphazard way of testing, and even under the most carefully guarded conditions there is a doubt about the utility of this neat test. It would be a mistake to place much reliance upon a system of testing that will occasionally give a good result by neat tensile tests when the sand test gives a poor one, and similarly when the neat

tensile strength test gives a poor result, and the sand test quite the opposite.

In the case of one tester known to the author, who has made up a considerable number of tests of neat cements, and who has had years of application at this work, it has been admitted that to fully understand the vagaries of neat cement testing demands a life-long study, and the more persistently a satisfactory conclusion is attempted in the judging of cements by the neat tensile test results, the more complicated grows the knowledge to be obtained, as time goes on.

Part of this **variation** is, however, inevitable. The strength of Portland cement depends so largely upon the details of the method of testing, that variations in manipulation among a number of other and minor causes make an appreciable difference in the results, and the neat tensile test is therefore governed by many circumstances.

The following particulars include perhaps most of the common causes of divergent results in the tensile test :—

(1) *Due to the Cement.*—Whether the cement is fresh or has been aerated, and if the latter, the period of aeration.

(2) *Due to Gauging.*—The amount of water used in mixing. The quality, character and temperature of the water used in gauging. The temperature of the room. Whether the cement is hand- or machine-mixed. Whether a non-porous material is used as a mixing tray.

(3) *Due to the Preparation of Briquettes.*—Whether a skilled operator has been employed. Whether the mould is of wood or iron. The shape of the mould, the method of filling, and whether the mould was filled at one or at several operations. Whether all air bubbles have been eliminated. Whether the mould is shaken, tapped rammed or pressed to make the briquettes of various densities.

Whether the briquettes are kept damp during setting, and whether kept dry or in water during the period of test. Whether the briquettes are made by the same operator on the same day under the same conditions. The temperature of the water used for immersion.

(4) *Due to the Testing.*—The temperature of the testing room. The length of time the briquettes have been

out of the water previous to testing. The area of the breaking section. The form of the briquettes and the shape of the cross-section at the point of fracture. The nature of the strain, and whether suddenly or gradually applied, and the time occupied in applying the strain. The form of clips in the testing machine for holding the briquettes. The direction of the strain as regards the breaking sections of the briquettes and the preventing of cross strains. The unequal bearings of the clips on the briquettes.

The fact that there are so many separate factors connected with the making and testing of briquettes, all of which have a distinct and definite influence on the subsequent tensile strength need not, however, unduly point to the inconsistency of the test.

For instance, the age of the cement after grinding is found to affect the tensile strength of a briquette; but as the manufacturer is responsible for the state in which the cement leaves his works, we can rely upon him looking after his own interests in this direction.

The percentage of **water** to be used in the gauging of neat briquettes is generally from 18 to 25, a proportion which varies according to the age, fineness and activity of the sample.

For briquettes of three parts sand to one of cement, about 8 per cent. of water is required (the percentage being calculated on the total weight of the sand and cement).

The best results are obtained with a minimum quantity of water, and a dry or stiff plastic mixture gives greater uniformity.

The water to be used in gauging must be clean and fresh, and of a temperature of about 60° to 70° Fahr. In the gauging of neat briquettes, the cement and water must be *thoroughly* mixed for one minute, and then pressed into the moulds by the trowel used in gauging.

Any excess cement should be removed and the top of the briquette smoothed off level with the mould. Particular care should be taken that the mixing is carried out upon some non-absorbent substance, such as a slab of slate or thick glass.

It should further be pointed out that the accurate test-

ing of cements for tensile strength is *not* a simple process. Some experience is necessary before the materials can be manipulated so as to obtain even approximately accurate results.

The tests carried out by inexperienced though intelligent and careful persons are usually very contradictory and inaccurate, and no amount of experience can totally eliminate the variations introduced by the personal equation of the operator.

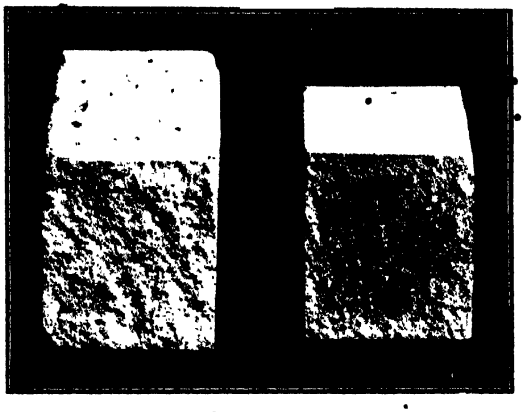


PLATE NO. I

An instance of such variation is shown in the illustration which is here given (plate No. I), showing two sections of two broken briquettes made from the same cement by two different operators. One briquette (containing air-holes) tested at 320 lbs. per square inch at seven days, and the other at 580 lbs.

The British Standard Specification requires that in testing Portland cement for its tensile strength, both neat and sand results shall be ascertained, the test being carried out as follows :—

Test For Tensile Strength (Neat Cement).

The breaking strength of neat cement shall be ascertained from briquettes of the shape described.

The briquettes shall be prepared in the following manner :—

The cement shall be mixed with such a proportion of water that the mixture shall be plastic when filled into the moulds used for forming the briquettes.

The cement, gauged as above, shall be filled into moulds of the form required to produce briquettes of the shape shown, each mould resting upon a non-porous plate. In filling the moulds the operator's hands and the blade of the ordinary gauging trowel shall alone be used. The trowel shall weigh about 7½ ozs. No ramming or hammering in any form will be permitted, nor shall any other instrument or apparatus other than the trowel before described be employed for this operation. The moulds after being filled may be shaken to the extent necessary for expelling the air.

Clean appliances shall be used for gauging, and the temperature of the water and that of the test room at the time the above operations are being performed shall be from 58 to 64 degrees Fahrenheit.

The briquettes shall be kept in a damp atmosphere for 24 hours after gauging, when they shall be removed from the moulds and immediately submerged in clean, fresh water, and left there until taken out for breaking. After they have been so taken out and until they are broken the briquettes shall not be allowed to become dry. The water in which they are submerged shall be renewed every seven days, and shall be maintained at a temperature of between 58 and 64 degrees Fahrenheit.

The briquettes shall be tested for breaking strength at 7 and 28 days respectively after gauging, six briquettes for each period. The breaking strength shall be the average tensile breaking strength of the six briquettes for each period. The briquettes to be tested shall be held in strong metal jaws of the shape shown, and the load shall be steadily and uniformly applied, starting from zero, and increased at the rate of 100 lbs. per square inch of section in 12 seconds.

The breaking strength of the briquettes at 7 days after gauging shall be not less than 450 lbs. per square inch of section.

The breaking strength of the briquettes at 28 days after

gauging shall show an increase on the breaking strength at 7 days, and shall be not less than the number of pounds per square inch of section arrived at from the following formula :—

$$\text{Breaking strength at 7 days} + \frac{40{,}000}{\text{Breaking strength at 7 days.}}$$

Test For Tensile Strength (Cement and Sand).

The breaking strength of cement and sand shall be ascertained from briquettes also of the shape shown in illustration. The briquettes shall be prepared in the following manner :—

A mixture of cement and sand in the proportion of one part by weight of cement to three parts by weight of the standard sand specified shall be gauged with sufficient water to wet the whole mass throughout without any excess of water being present.

The mixture, gauged as above, shall be evenly distributed in moulds of the form required to produce briquettes of the shape shown in illustration, each mould resting upon a non-porous plate. After filling a mould a small heap of the mixture shall be placed upon that in the mould and patted down with the Standard Spatula, until the mixture is level with the top of the mould. This last operation shall be repeated a second time, and the mixture patted down until water appears on the surface; the flat only of the Standard Spatula is to be used and no other instrument or apparatus is to be employed for this operation. The mould, after being filled, may be shaken to the extent necessary for expelling the air. No ramming or hammering in any form will be permitted during the preparation of the briquettes, which shall then be finished off in the moulds by smoothing the surface with the blade of a trowel.

Clean appliances shall be employed for gauging, and the temperature of the water and that of the test room at the time the above operations are performed shall be from 58 to 64 degrees Fahrenheit.

The briquettes shall be kept in a damp atmosphere for 24 hours after gauging, when they shall be removed from the moulds and immediately submerged in clean, fresh

water, and left there until taken out for breaking. After they have been so taken out and until they are broken, the briquettes shall not be allowed to become dry. The water in which they are submerged shall be renewed every seven days, and maintained at a temperature of between 58 and 64 degrees Fahrenheit.

The briquettes shall be tested for breaking strength at 7 and 28 days respectively after gauging, six briquettes for each period. The breaking strength shall be the average tensile breaking strength of the six briquettes for each period. The briquettes to be tested shall be held in strong metal jaws of the shape shown, and the load steadily and uniformly applied, starting from zero and increased at the rate of 100 lbs. per square inch of section in 12 seconds.

The breaking strength of the briquettes at 7 days after gauging shall be not less than 200 lbs. per square inch of section. The breaking strength of the briquettes at 28 days after gauging shall show an increase on the average breaking strength at 7 days, and shall be not less than the number of pounds per square inch of section arrived at from the following formula :—

$$\text{Breaking strength at 7 days} + \frac{10{,}000}{\text{Breaking strength at 7 days.}}$$

The standard sand shall be obtained from Leighton Buzzard, be thoroughly washed and dried, and shall pass through a sieve of 20 by 20 meshes per square inch, and be retained on a sieve of 30 by 30 meshes per square inch. The sieves shall be prepared from wire-cloth, the wires being .0164 inch and .0108 inch in diameter respectively. The wire-cloth shall be woven (not twilled), the cloth being carefully mounted on the frames without distortion.

In the **preparation of briquettes** it is advisable, as far as possible, to have all moulds (which should be made of gun-metal) shaped exactly alike, and the gauging of each briquette should be carried out in exactly the same manner. As the value of the tensile strength test depends mainly upon the preparation of the briquettes, it is necessary that precautions be taken so that the personal equation is, as far as possible, eliminated, by ensuring that the proper percentage of cement and water is always strictly observed. In preparing the briquettes, after the mixing

of the samples of cement, care should be taken that the moulds have been properly cleaned and placed on small plates of iron or slate.

For the neat test, weigh the registered quantity of cement for each briquette and turn out the weighed cement in a heap on to the slate slab, and, after hollowing out, gradually pour on the exact amount of water which it has been previously determined will provide the consistency required according to the standard specification.

Then trowel the cement thoroughly and quickly, and fill the mould so that the material is solid and free from air spaces, and finally trowel the top side of the briquette.

During setting, the briquette should be placed in a damp place or covered with a cloth saturated with water, and allowed to remain for 24 hours in a place free from any vibration. A briquette that has thus been allowed to harden slowly, and has been kept free from any hot, dry, or draughty atmosphere, may be relied upon as being stronger, and the testing results more uniform than is the case where the water is evaporated from the cement too quickly.

When the briquettes are thoroughly set they should be immersed in water until ready for testing.

Some of the **testing machines** generally used in this country are illustrated here, the "Adie" testing machine, with the automatic regulator, being, perhaps, more often employed.

The instructions for working the "Adie" testing machine are as follows :—

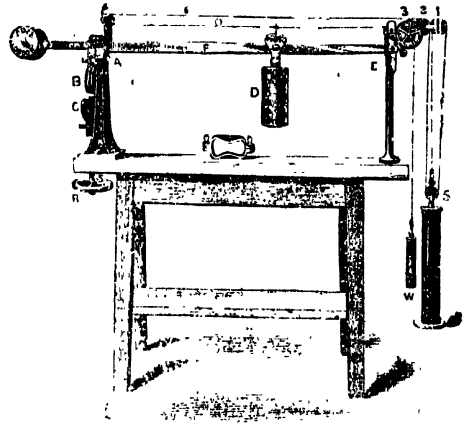
Fix the forked pillar E by means of nut and washer, drop the spindle S into its place in the table, then put the standard A in position and insert the notched plate in groove of spindle, bolting down A so that the lever when strained (by putting a moulded briquette of cement into the clips B and C, and then tightening by means of the wheel R) may take its position freely in the centre of forked pillar E. Put the lever F in its place so that the knife edge rests in groove of hardened steel on top of standard A; hang on clips B and C; put head weight in place and fix by thumb screw, and the machine is then ready for use.

To test the briquette, insert in the clips B and C, then turn the wheel R until the lever at E rises well above the

zero line; then by turning handle on standard E the weight is moved gently along until the briquette has broken, the strain being then read off on lever.

With machines having two rows of figures, use the bottom row with head-weight in its place on the lever, and the top row with head-weight and thumb screw off.

The automatic regulator has been designed to give a uniform rate of travel to the weight, so that all tests are made at an equal number of lbs. per minute; but this can



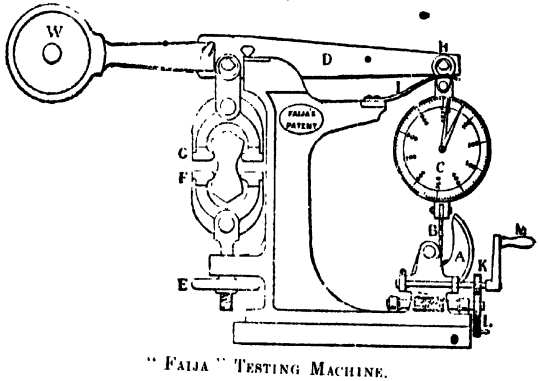
"Adie" Testing Machine.

be altered to any speed required by means of a cock in piston; it is also self-acting, and when the test is started, needs no interference whatever, and directly the briquette breaks the weight is held fast by means of clutch or brake until released.

To fix the automatic regulator, fasten the brass tube to floor underneath the pulley marked 2, so that the weight W just clears, and fill the tube with clear lubricating oil, attach the long cord to left-hand side of vernier, pass round pulley 4 on top of pillar A, and back and down over pulley 2, under pulley 5, and upwards round No. 1, and

down again to eye at 5, attach the short cord to right-hand side of vernier, pass it over and twice round the pulley to prevent slipping.

To use automatic regulator, see that the vernier is at the commencement of scale, then place briquette into its place, tighten by the wheel R until the strain is sufficient to lift the lever well above the zero line on standard E, then hang on brass weight W, which will at once commence to move vernier along the lever; see that the vernier weight does not rock while travelling. To commence another test, remove weight W, lift lever by means of lifter, which is fixed



"Faija" Testing Machine.

to standard E, then press on the cross handle at top of piston rod, which will let piston down gently, and return vernier to commencement of scale, and proceed as already described.

In continuing with the testing machines used in this country, reference should be made to the **"Faija" apparatus** as being one commonly used by manufacturers and engineers, and a description of the working of this machine will no doubt be useful for reference.

To use the "Faija" machine:—See that the quadrant A is in the position shown in sketch so that the chain B to the dial C is slack, and the lever D free and balanced.

Turn the wheel E from right to left, until the lower clip F can be raised into contact with the upper clip G.

Insert the briquette to be tested in the clips, taking care that it is put in evenly, so that the pull is true and vertical, then turn the wheel E from left to right, which will bring down the lower clip F, and secure the briquette firmly in the clips. (It is generally advisable to put such a strain on the briquette by turning wheel E that about 100 lbs. is indicated on the dial.) When in this positon there should be about half-an-inch between the under side of knife edge H, and the buffer or recoil spring I.

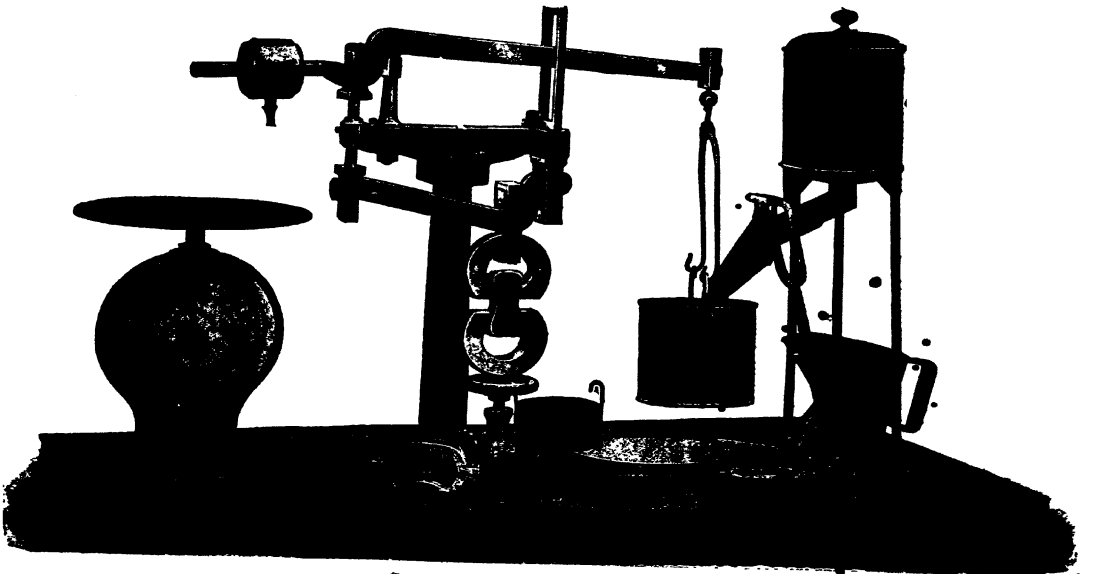
Having seen that the pinion K is in gear with the wheel L, turn the handle M until the briquette breaks. The loose pointer will show on the dial the strain in lbs. at which the briquette broke.

To return to zero :—Throw the pinion K out of gear with the wheel L by turning the pin and pushing the spindle to the left; turn the wheel L from left to right until the quadrant A has returned to its normal position with the chain B slack; put the loose pointer back to zero: release the lower clip F by turning wheel E from right to left; remove the broken briquette, and insert the next that is to be broken.

Another useful apparatus for testing the tensile strength of Portand cement is **Salter's Cement Testing Machine.**

This apparatus has now been improved and revised so as to meet the requirements of the British Standard Specification for cement testing, and it will be found as useful a machine as can be devised for the purpose. It has also been increased in strength, and the bucket shot-run, and scales made suitable for weights up to 1,200 lbs., as owing to the recent improvements in the manufacture of cement it is no unusual thing to find neat tensile tests up to 900 to 1,000 lbs. on the square inch at seven days, and, of course, still more than that at longer dates.

The shape of the clips is in accordance with Standard requirements, and these are sufficiently massive to require no cross stays. As the speed of the shot feed is now standardised, a can for hand feed is not supplied. The speed of the shot may be adjusted to 450 or 500 lbs. per minute. With the former speed, as the bucket indicates 50 lbs., it makes up a total load of 500 lbs. on the briquette in the



SALTER'S CEMENT TESTING MACHINE.

first minute. It is better to use this speed with a sand briquette, but if the briquette is neat and the test is likely to be considerably over 500 lbs., it had better be set to the speed at the 500 lbs. mark.

Standard briquette moulds are also supplied with the apparatus, and a glass frame to keep the machine clean and bright. The machine should be well looked after, and not allowed to get into a dirty condition, or its reliability may be impaired.

The apparatus consists of a japanned cast-iron column, carrying a pair of compound levers, having a combined leverage of 50 to 1. The levers are fitted with tempered steel knife edges, which rest on polished concave bearings, also of tempered steel, thus obtaining a very sensitive balance. A sliding balance weight for the purpose of setting the levers in equilibrium is fitted to the upper lever. The upper clamp, to receive the cement briquette, is suspended from a knife edge on the lower lever; the lower clamp is attached to the base of the column, and is adjustable by means of a screw and a small hand wheel. The supply of shot to the bucket is automatically cut off at the moment the briquette breaks.

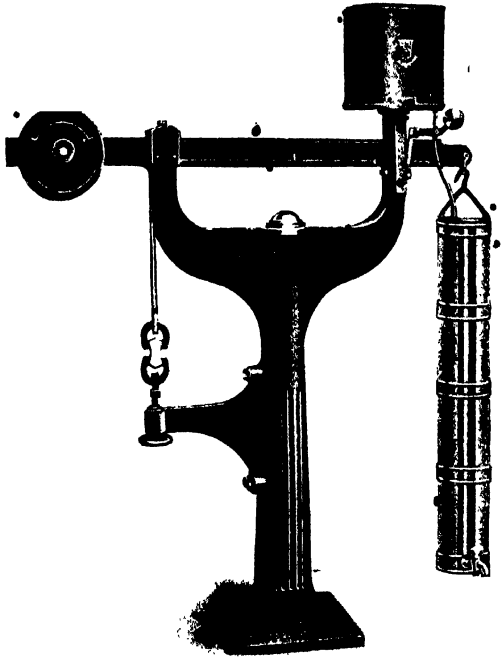
To use the machine, set the levers "floating" by means of the sliding balance weight. Once set tightly, this need not be touched. If, however, it is desired to start the load from zero, this weight can be adjusted to balance the weight of the can by running it out to the end of the lever. In that case either 50 lbs. must be deducted from the scale when weighing the shot and can, or, to save this calculation, the pointer of the balance can be adjusted by the screw for that purpose so as to stand at zero with the weight of the can, and in that case the pointer will give the weight of shot only.

The briquette is next removed from its tank, wiped with a cloth, and placed in the clamps, centrally and flush with the sides.

Hang the bucket on the bridle and screw up the hand wheel until the top side of the lever from which the shot can is suspended is about half an inch from the top of the guide. This then allows ample downward movement, if the clips slide up the briquette as the load increases, and prevents the lever touching the bottom of the

guide before rupture, and avoids the necessity of stopping the machine to screw up the wheel again.

As soon as all is ready and all the knife edges are seen to rest square in their bearings, depress the handle to the



Avery's Cement Tester (with Water).

first notch. If starting from zero the shot should be adjusted to run at the 500 lbs. per minute.

As soon as the briquette breaks, the bucket falls on to the rubber pad and depresses the lever by which the shutter is released, and so cuts off the supply of shot. All shot must be the same size—No. 6 for preference.

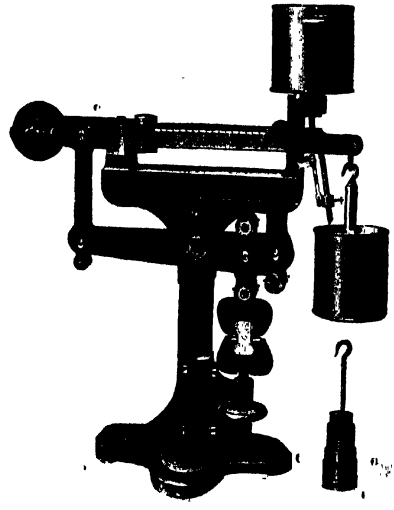
The bucket is then removed and placed on the spring

balance by which the breaking load is at once indicated without calculation.

If by any chance the shot should hang up from any cause, the container should be tapped.

The apparatus will test with equal correctness any load from ¼ lb. up to 1,200 lbs.

After use the machine should be at once rubbed down with a dry, soft cloth, and put under cover.



AVERY'S "TABLE PATTERN" CEMENT TESTER (WITH SHOT).

Other machines very commonly used for testing cement for tensile strength are the **Avery's Table Pattern Cement Testers** as illustrated.

These tests are applied by means of fine shot flowing into a receiver at the end of the steelyard, or in another apparatus water is used to apply the strain. This ensures an even and steady application of the strain by which means accurate results are obtained.

In the former machine the flow of the shot is so arranged

that the strain is applied to the specimen at a uniform rate of increase of 100 lb. in 12 seconds.

When the specimen fractures, the shot is automatically cut off by a trigger operated by the sudden fall of the steelyard.

The breaking strain of the specimen is computed from the weight of the shot by unhooking the receiver and suspending it from the other end of steelyard. The receiver is then weighed on the steelyard, which gives the strain (in lbs.) at which the specimen was fractured, and avoids the use of a separate weighing machine.

The machine is strongly constructed, the standard steelyard and levers being of cast-iron, and the knife edges and bearings throughout of hardened steel.

New pattern self-adjusting gun-metal grips are provided for holding the briquettes, designed so that the strain is applied in true alignment, thus preventing any tendency to a lateral strain, which would "tear" the briquette and give inaccurate results—the grips agree with the Standard Specification.

In regard to the tests of **ageing** in cement and concrete mixtures and their effect upon the tensile strength results some interesting and useful figures are published by the Geological Survey Department of the United States.

A report upon "Portland Cement Mortars and their Constituent Materials" gives the results of over 25,000 tests made during the years 1905-7 at the Structural Materials Testing Laboratories at Forest Park, St. Louis, Mo. The report may be divided into two parts. The first deals with tests, chemical and physical, of seven different cements, and of a typical cement composed of a mixture of equal parts of each of the seven brands; and of tests on 1 : 3 mortars made with these cements in which a standard sand was used. The second part of the report deals with tests of 22 sands, 12 gravel screenings, and 25 stone screenings, and of mortars made with these sands, and with the gravel and stone screenings.

The tests of the seven cements and of the mix indicate clearly that the **maximum tensile strength** of specimens made from **neat cement** is reached in about ninety days. From that time till 180 days the tensile strength remains nearly stationary, and from 180 to 360 days there

is a decrease in tensile strength of about 25 per cent. In the case of standard mortars made from the various cements all showed the same characteristic falling off in tensile strength from 180 to 360 days. The mixture, however, showed a falling off from 90 to 180, but a slight increase in strength from 180 to 360. Further tests made on the mixture, after storage in air-tight cans for periods varying from a few weeks to a year, indicated that after 180 days the tensile strength was fairly constant or slightly increased. Altogether, the tests for tensile strength on both specimens made from neat cement and from standard mortars do not exhibit the uniformity which might be expected. As regards the falling off in tensile strength from 180 to 360 days, these tests indicate the necessity of carrying tests over a further period of time, and also of making tests at a 270 day period. The tests on the compressive strength indicate a steady and rapid increase in strength up to 180 days and a slight increase in strength from then onwards. Tests on the **transverse** strength showed an increase up to 90 days, practically stationary from 90 up to 180 days, and a slight decrease from that time to 360 days.

A study of the percentage of gain in strength for various periods of time discloses the important fact that while cements may test high or low at seven days, at 360 days all are fairly close to each other. Cements which tested low at seven days, as a matter of fact tested higher at 360 days than did those giving high strengths at seven days.

The tests indicate, further, that mortars made with a cement composed of a mixture of a number of standard brands are likely to prove more efficient than mortars made with any of the individual brands. Further tests, however, must be made before this hypothesis can be definitely established.

The tests made on the sands, gravel, and stone screenings indicate that the tensile strength of mortars made from them decreases with an increase in the percentage of voids. The same is true for the compressive and transverse tests, though not so marked in the tests for transverse strength. Tests for **density** of mortar indicate, as would be expected, that the density is greatest for the least percentage of voids.

The New York Public Service Commission, in 1905,

commenced an investigation of the comparative tensile strength of neat cement and cement and sand briquettes at varying periods up to two years. The results of the investigation were read at a meeting of the American Association of Portland Cement Manufacturers, in December, 1907, and are very instructive reading. The investigation primarily had reference to the theoretical chemical composition of Portland cement; but as the tests were very exhaustive and have the advantage of being original, we propose to examine them so far as they relate to the **ageing of concrete.**

In all, fourteen different brands of Portland cement were taken, and nine tests from each brand were made, so that the figures in the following tables show the results of 126 tests. The briquettes were broken at seven days, twenty-eight days, three, six, twelve months, and two years. Five different mixtures were tested at each of these periods. After averaging all the tests of each of these mixtures, the results are given in Table I.

TABLE I.—TENSILE RESULTS OF NEAT CEMENT AND CONCRETE AT DIFFERENT PERIODS.

Average Results of Seventy-two Tests.

	Age of Samples (lbs. per square inch)					
	Seven Days	Twenty-eight Days	Three Months	Six Months	Twelve Months	Two Years
Neat Cement ...	687	785	758	777	795	763
Standard Sand 2 and 1 ...	387	502	520	509	497	400
Natural Sand. 2 and 1 ...	411	535	593	630	660	697
Standard Sand. 3 and 1 ...	245	356	377	370	358	344
Natural Sand. 3 and 1 ...	336	457	538	556	590	608

When the above results have been plotted into curves it will be seen that neat cement reaches practically its maximum strength in the very short period of twenty-eight

days, and in the large majority of cases was found to decrease at three months and onwards. Quartz sand concrete, however, did not reach its maximum until three months, after which the strength fell off. The American standard crushed quartz sand consists of that portion rejected on a sieve of twenty meshes per lineal inch. That it is decidedly inferior to the natural sand used in the above tests is doubtless due to the respective size of the grains. Any advantage which the natural sand may have through being slightly clayey would not account entirely for the higher tests.

The period of maximum maturity is again postponed with the natural sand; indeed, there is a steady gain throughout the whole of the periods of tests. That this gain is relatively small, however, may be better seen from Table II., in which all the results have been proportionately reduced so that the tests at six months are represented by the figures 100, and all the other tests of the same mix reduced in the same proportion.

TABLE II.—PROPORTIONATE TENSILE RESULTS AT DIFFERENT PERIODS.

	Age of Samples					
	Seven Days	Twenty-eight Days	Three Months	Six Months	Twelve Months	Two Years
Neat Cement ...	38	101	97.5	100	102	98
Standard Sand, 2 and 1 ...	76	98	102	100	98	96
Natural Sand, 2 and 1 ...	65	85	94	100	105	110
Standard Sand, 3 and 1 ...	66	96	102	100	97	93
Natural Sand, 3 and 1 ...	80	82	97	100	106	109

Neat cements are, therefore, practically as strong at twenty-eight days as at two years, while of concrete mixtures the natural sand samples show more steady increase. For the first three months the increase would be from 25 to 50 per cent. of the seven days' test; the second three months may bring about an increment of 5 or 6 per cent., or in some cases none at all, while after six months the quarterly periods will probably see an increment of 1 or 2 per cent., or may even show retrograde movement in the hardening process.

A further study of the figures in Table I. will show that there is a much more satisfactory way of **hardening concrete** than by ageing. The natural sand tests, which are assumed to approach more nearly to actual practice than do the others, show that at three months a 2 to 1 mixture is as strong as a leaner 3 to 1 mixture at twelve months, and has the advantage that the increased strength is permanently retained. In the manufacture of paving slabs and reinforced concrete beams it will probably be found that strength can be more economically obtained by increasing the Portland cement content and shortening the period of maturity. By this means the concrete is as strong or stronger than the low cement and older samples, and has the additional advantage that the short-period concrete will continue gaining strength where the older material has perhaps attained its maximum.

Some results of investigations as to the relation which the **proportion of cement** bears to the ultimate strength of the concrete have been published in "Ciments et Chaux Hydrauliques." In Table III. are given the results of tensile tests, at one year, of briquettes containing varying proportions of cement to one volume of sand. These results represent the average of a very large number of tests, and the proportions of cement given in the first column were taken by weight, assuming the sand to weigh 100 lbs. per cubic foot. For convenience of comparison, columns are added in which the most advantageous strain is represented by the figure 100, and all other results reduced proportionately. Within certain limits the strength of sand and cement concrete varies practically with the amount of cement used, and the addition of, say, 10 per cent. of cement will probably increase the strength

in a like proportion. For instance, a reference to Table III. shows that if 100 parts of cement give a tensile strain proportionately represented by 100, the addition of 25 parts of cement increases the tensile strain to 129, and similarly 219 parts show a proportionate result in strength of 212, or practically in direct proportion to the proportion of cement used. This is nearly true of the tensile strain, while as regards crushing strain the advantage of the richer mixtures is even more pronounced. For instance, upon referring to Table III., it will be seen that with the proportion of .5 cement, or, say, 2 and 1 sand and cement, the tensile strength ceases to maintain its proportionate increase, although it still increases its strength with richer mixtures.

TABLE III.—TENSILE AND CRUSHING STRAINS OF CONCRETE ONE YEAR OLD.

Volume of Cement to 1 of Sand	Tensile Strain lbs. per sq. in.	Crushing Strain lbs.	Proportion of Equivalents		
			Cement Volume	Tensile	Crushing
.156	212	1730	71	63	58
.218	337	2980	100	100	100
.285	435	4020	125	129	135
.345	499	5580	151	145	187
.4	594	5820	175	176	195
.5	713	7750	219	212	260
.625	767	7670	274	228	256

Compression and Transverse Tests.

CHAPTER XXIII.

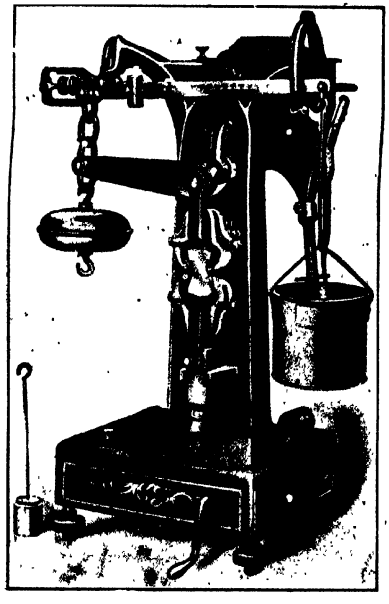
FOLLOWING the impetus given to the consumption of Portland cement for the purposes of concrete construction generally, and reinforced concrete in particular, the interest in the question of compression tests for concrete has of late years been considerably strengthened.

Until the advent of reinforced concrete, the poorest of mixtures could generally be relied upon to withstand any reasonable compressive stress to which its use would be likely to render it subject. The source of weakness in the material which had to be guarded against was then its tensile strain, and inasmuch as the compression strength of cement was known to be far in excess of its tensile strength, there was very little call for a test which was seldom necessary and difficult to apply.

In the matter of neat cement tests the tensile strain is still the one upon which reliance is placed in ascertaining the quality of the principal ingredient of the concrete. It is rarely necessary to determine the compressive strength of neat cement, as such a test would give no indication of the quality of the concrete which is to be used in a specific work. There are so many factors which determine the strength of this material that the only tests of value must be made with the actual ingredients which will be used in the work to be carried out. As far as possible it is also of importance that the damping and mixing of the ingredients should be conducted precisely as will occur in the work for which the concrete is to be used.

Crushing tests may be taken with one or other of the ordinary briquette testing machines, provided with compression plates. The illustration shows an attachment

which may be used for a compression test with **Fairbank's** tensile testing apparatus. The capacity of such an arrangement is, however, limited, and for larger cubes of four and six inch sides a special crushing machine is generally used. Although rarely carried out in this country by cement manufacturers, the test for compression or crush-



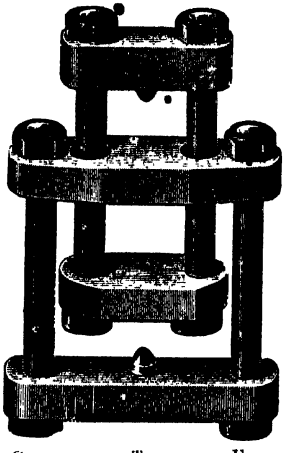
FAIRBANK'S TENSILE TESTING MACHINE.

ing is commonly performed in factories on the Continent, a block of cement and sand being used about $2\frac{3}{4}$ inches by $2\frac{3}{4}$ inches by $2\frac{3}{4}$ inches, and prepared in a similar way to the briquettes.

In order to secure uniformity in the results of testing by compression, it is preferable to make use of some mechanical process for the preparation of the test blocks. Care must be exercised in arranging that the pressure be

even all over the test block, and, consequently, its two faces should be well trowelled and smooth, and all sand or grit removed.

The preparation of test blocks for compression is carried out on the Continent by well mixing 14 ozs. of cement with 42 ozs. of dry standard sand. To this is added 160 c.c. of



ATTACHMENT FOR COMPRESSION TEST WITH FAIRBANK'S TESTING MACHINE.

clean water, and the mortar well stirred. Assuming that these proportions be adopted, take about 30 ozs. of the mortar and fill into a cubical mould under pressure.

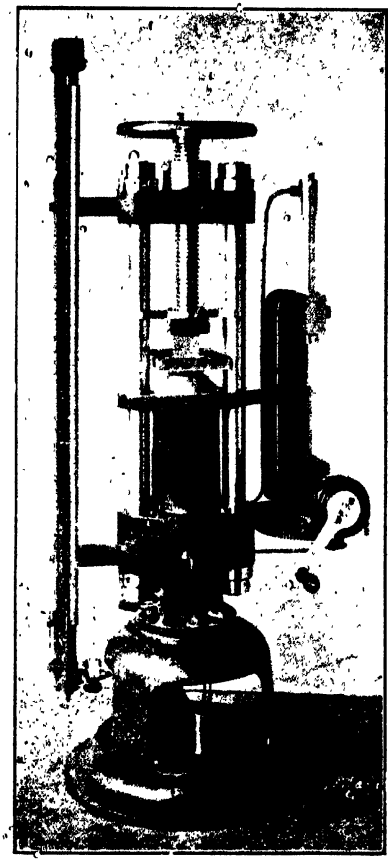
After the preparation of the block by this process, well trowel the two faces, and put aside to set for a period of 24 hours, after the expiration of which time, place the blocks in water. The immersion lasts until the test takes place, and each block must be tested immediately it is taken from the water.

The Standard machine for carrying out the compression tests is a hydraulic apparatus by Amsler-Laffon, of Schaffhausen, Switzerland.

The machine tests up to 60,000 lbs. compression.

In testing the cement cubes by this instrument, the lower compression plate rests with a spherical surface on the piston contained in the main cylinder. The upper com-

pression plate just touches the flat end of the screw spindle, which is only used to set the distance between the plates to the size of the test block.



Amsler-Laffon Apparatus for Compression Test.

During the test the upper plate is at rest, whilst the lower plate is raised by the piston pressing upwards under the

action of oil coming from the plunger pump, which is driven by a handle shown at the right of the illustration.

The pressure of oil in the main cylinder multiplied by the sectional area of the piston is equal to the strain on the specimen block, provided that the pistons move without friction, and this has been arranged in the construction of the apparatus.

The oil also assists to well lubricate and tighten the piston.

The pressure exerted acts at the same time upon a thin piston transmitting its movement to a lower piston which pushes the mercury into the transparent tube of the gauge on the left of the machine. The mercury rises here to such a height that the pressure of the column of mercury balances the pressure of the piston.

In this way, the rise of mercury is a means of measuring the pressure of the main points.

The accuracy of the results read off the gauge depends only on the proper working of the pistons in their cylinders. This can be controlled at any time by moving the pistons with the hand and observing that all move smoothly. As there is neither stuffing box nor other lining for tightening the pistons, the friction will be smaller rather than greater when the machine is loaded, because of the slight widening of the cylinders.

The machine will improve by frequent use in consequence of the wear of any roughness on the cylinders and pistons.

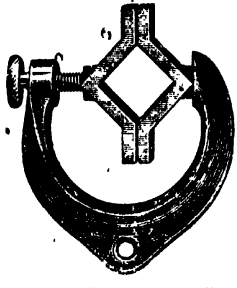
As regards accuracy, the machine is, at least, equal to any good lever machine; in respect to wear and tear, it is almost indestructible.

No special foundation is required for the machine, save only that the floor should be strong enough to carry the machine safely.

An English machine for carrying out this test has been placed on the market by Messrs. **W. G. Bailey & Co.,** Ltd., of Manchester, and is here illustrated. This is a hydraulic press in which the load is increased by a screw plunger worked by hand. The cube is fixed tight between the compression plates by the adjusting screw shown, which is also worked by hand. The load on the specimen is given at once by the position of the pointer of the pressure gauge, which is arranged to retain its maximum

position after the specimen has failed by crushing. The fluid used in this machine is glycerine or oil.

As previously explained, a very important point to be noted in testing cubes by compression is the manner in which the specimens are bedded on the compression plates. It is necessary that the distribution of the load over the top and bottom surfaces of the block should be quite uniform. The most satisfactory way of **bedding** the **cubes** is by using plaster of Paris. The plaster is laid to a thickness



Mould for Compression Blocks.

of about a quarter of an inch on the plates, which should be oiled to prevent the plaster sticking. The block is bedded on the plaster before the latter is set, and the test may be taken about ten minutes after both top and bottom faces of the cube have been so treated.

The shape of the fractured specimen is the usual one of two more or less perfect pyramids joined together at the apex.

Neat cement tested after seven days may be expected to provide a crushing strain of 5,000 lbs. per square inch, and about 6,000 lbs. in 28 days. The strains which result from a mixture of cement and sand, or cement, sand, and stone, vary within very wide limits with the following conditions :—

The amount of water used in mixing.

The thoroughness with which the mixing is conducted.

The amount of cement used.

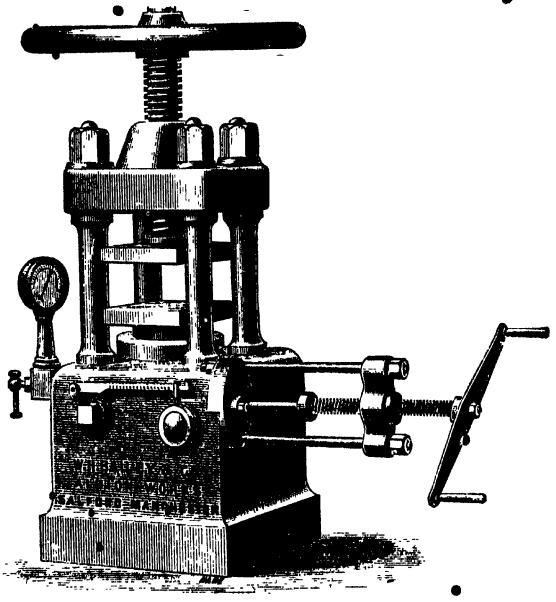
The size and shape of the grains of sand.

The hardness, shape, and size of the stone forming the aggregate.

The age of the specimen.

In ordinary commercial machine-mixed concrete made with four parts by measure of crushed hard rock or gravel which will pass through a one-inch sieve, two parts of clean sand and one part cement, a crushing strain of from 2,400 to 3,000 lbs. per square inch would be obtained in one or two months.

Hard limestones, trap rock, or crushed granite form suitable aggregates, so far as compressive strains are concerned.



Bailey's Testing Machine for Compression Cubes of Cement.

Gravel also gives satisfactory results, although the fact that gravel lacks the sharp, angular formation of crushed stone may cause it to give inferior results in short-time tests. Many concrete engineers of experience regard gravel as giving quite as high results in compression as crushed stone, notwithstanding its rounded and smooth form.

A mixture of sand and cement in equal proportions, tested after two and a half years' setting, has been found

to crush at 11,330 lbs. per square inch, while a 2½ and 1 mixture at the same age gave a result of 8,610 lbs., according to tests taken at the Watertown Arsenal, U.S.A.

Among the highest results obtained in ordinary commercial concrete are those advertised by the various makers of paving slabs in England. Most of the slab manufacturers publish properly authenticated tests, giving strains from 8,000 lbs. per square inch to nearly 13,000 lbs. at three months. These results were obtained in granite concrete consisting for the most part of ¾-inch chippings and cement in the proportions of 2½ and 1 or 3 and 1. The concrete is obviously factory-made, and machine-mixed, and such high results cannot be looked for in work carried out *in situ*.

Some tests are here given of concrete as used in connection with the King's Dock, Swansea, which afford a clearer idea of the results obtained under ordinary conditions in civil engineering work.

TABLE OF TESTS MADE WITH CUBES OF 1 : 1¾ : 2¾ CONCRETE.
SIZE OF TEST BLOCKS—6IN. CUBE.

Description	Weight	Age	Crushed at	
			Per sq. inch	Per sq. foot
1. Rotary kiln cement —	Lbs.	Months	Lbs.	Tons
Aggregate, granite chippings and coarse sand. Average of six tests	18.85 18.73 18.72	3 6 9	3,685 3,998 4,467	237.0 257.1 287.3
2. Rotary kiln cement—				
Average of two tests ...	16.69 16.39	3 6	2,507 2,453	161.2 157.8
Aggregate, natural fine gravel and coarse sand. Single tests	16.63 15.83	9 9	2,972 1,940	191.1 124.8
3. Ordinary kiln cement —				
Aggregate, natural fine gravel and coarse sand. Average of two tests	15.92 15.33 15.55	3 6 9	1,182 1,440 1,541	76.0 92.6 99.1

In practical reinforced work there should be no difficulty in securing that the concrete will sustain a crushing stress of 2,400 lbs. per sq. inch, and on this basis the rules of most countries and governments require that the working stresses on concrete shall not exceed 600 lbs. per square

inch in beams and 500 lbs. in columns to provide an ample margin of safety.

With a good cement and ordinary care there should be no danger in working to these figures.

An instance of the strength of concrete made from rotary kiln cement may here be cited as of interest. During the demolition of a concrete wall in connection with the Stockport water scheme a portion of the concrete was set aside, and handed over to testing experts for examination. Cubes were cut from the sample and tested, the average of the tests showing 4,150 lbs. per square inch, and the highest being 5,039 lbs. The concrete was about two years old, and was made with limestone chippings 0 and 1, and formed part of the ordinary contractor's work as provided for a concrete dam. The samples were obtained by blasting.

The **ratio** between the **tensile** and **crushing** strains of concrete is not invariably constant. The compressive strength increases more rapidly with age than does the tensile strain. At two months the ratio of compression to tension is about 9. At six months it would increase to 10, and at twelve months to 10½, according to results published by Johnson in "Materials of Construction." A similar increase in the ratio with richer mixtures is also noticed by Candlot, who published in "Ciments et Chaux Hydrauliques" the results of tests of cement mortars which showed a ratio of compression to tension of from 7.2 to 10, varying with the richness of the mixture, and being greater with richer mixtures. We give below an extract from these tests converted from the metric system.

RATIO OF COMPRESSION TO TENSION WITH VARYING MIXTURES AT 12 MONTHS' MATURITY.

Lbs. of cement per cubic yard of sand	Tension in lbs. per sq. inch	Compression in lbs. per sq. inch	Ratio Compression Tension
250	70	507	7.2
420	212	1,730	8.1
590	337	2,980	8.8
750	435	4,020	9.2
930	490	5,580	11.4
1,100	594	5,820	9.8
1,350	713	7,750	10 9
1,700	767	7,670	10.0

Transverse Tests.—Although little is at present heard from cement manufacturers and engineers respecting the transverse tests of Portland cement and concrete, it must be admitted that this test is extremely useful in showing the hardening of cements between the final setting time and the usual periods for ascertaining their tensile strength. The cement maker has found the tensile strength test a convenient one to adopt at his works, where apparatus has been installed for taking the test. The engineer, now that reinforced concrete has become more general, has naturally a somewhat greater interest in the crushing test, at least so far as the testing of concrete is concerned.

The transverse test has, therefore, not received sufficient consideration, although for many years it has been held in esteem by clerks of works and others having the actual supervision and practical experience of concrete work. The chief reason, perhaps, is due to the ease with which such a test can be conducted on the works, and the latitude it allows in the way of varying dimensions of the specimens to be tested, but little information is available as to the further utility and results of the transverse tests.

In many cases, however, the transverse test is the only available method of testing, particularly with regard to concretes containing large size aggregate. A tensile test of one square inch in the usual testing machine is clearly impossible, while a crushing test involves the use of expensive apparatus.

A great advantage from the transverse tests is that a very simple arrangement of two knife edges upon which the bar is to rest, together with the means of applying the load to the centre of the bar, can be made to give reliable results. In the case where the weights applied are multiplied by a lever arrangement, care should be taken that the load is applied quite perpendicularly and at right angles to the axis of the bar.

The relation which the modulus of rupture or extreme fibre stress bears to the tensile strength of the material is somewhat inconstant. The usual formula :—

$$F = \frac{3Wl}{2bd^2}$$

gives higher results than could be obtained by the direct

tension test, and general experience would point to F representing from one-and-a-half times to twice the actual tensile strength.

The relation between the transverse test and tensile test of a given sample varies as much with regard to conditions as does the relation between the tensile and crushing tests. The modulus of rupture may, however, be expected to bear a proportion of from 1.5 to twice the tensile strength as previously mentioned, with the bar of neat cement to be tested, given a span of five and one-third inches with a cross section of two inches square.

It will be seen from the formula quoted that the breaking weight applied at the centre of such a bar will be the same as the extreme fibre stress of the specimen, and that a weight of 600 lbs. applied would denote a stress of 600 lbs. per square inch on the specimen. A multiplying lever would be required with these constants, and in some cases it would be found more convenient to provide a longer span, and thus reduce the weight necessary to break the specimen.

In testing concrete the section of the bar must depend to a large extent upon the size of the aggregate used, so that it is not possible for any large piece of stone to take up a considerable proportionate area of the point under tension.

In the ordinary way transverse tests at short dates are somewhat unreliable unless ascertained by a carefully-prepared tester, and particularly is this so with regard to concrete mixtures, and where variations of moisture and heat prevail. The bars are often immersed in water for a seven-day test to secure as much uniformity as possible, although at earlier dates and during the hardening of neat cements the transverse test is most valuable, providing a suitable apparatus is available.

A number of American tests, given by Sabin, show results in transverse strength with bars of the dimensions mentioned previously of from 1,100 to 1,300 lbs. per square inch at different periods as compared with 588 to 733 lbs. tensile strain.

A large number of tests have been made from time to time with bars of neat cement of one square inch section on a span of 6 inches, the bars being 8 inches in

length. An average breaking weight of 91 lbs. applied at the centre which was obtained would denote a theoretical fibre stress of 819 lbs. per square inch, as compared with a tensile strength of 450 lbs. per square inch obtained from the same cement. This result represents about 1.8 times the tensile strength, and agrees with the previous test quoted. It was found from the experiments then made that bars of neat cement made with 20 per cent. of water, 8 inches long and 1 inch square in section, bore a weight of one-fifth of the specified tensile strain without breaking after seven days' immersion in water. The span of the bars was 6 inches.

It has been suggested that a transverse test should be incorporated in cement specifications, and that this test would appear to have a greater sphere of usefulness in the testing of mortars and concretes, whose composition makes them unsuitable for making up into the small briquettes necessary for subjecting to the tensile test. In this connection the transverse test will certainly be found of value.

The test, however, has another important sphere of usefulness, and this was referred to in a preceding chapter on the setting of cement.

It has been stated that the difference in the times of setting, and of the ultimate hardening of numerous cements produced by the rotary kiln to-day, is a factor that must be correctly and surely gauged by extensive cement users so as to avoid untoward consequences in concrete work due to the delay sometimes experienced in the hardening of concretes after being placed in position.

It is, for instance, far more important for the contractor to know how long he might work his cement without damage than how long it takes to obtain a certain arbitrary tensile strength.

The difference in the times of hardening of various cements can be accurately gauged by the use of the testing machine as devised by the author and the results of such tests upon many cements at present on the market explain in what way the transverse test demonstrates the hardening of cements at early dates and the important bearing this has upon the strains to be applied to "green" concrete made with certain slow hardening cements. These tests should all be made with the neat

cements, for it is assumed that in appraising the setting and hardening qualities of the cement to be used the results from the neat cement will not be materially altered other than in proper ratio, by the addition of aggregates used in the making of concretes with such cements.

The study of neat transverse tests shows, for instance, the time and dates at which the hardening of certain cements can be stated to have arrived at maturity, and this is important information in the uses of cement in view of the serious results which may arise from an inordinately slow setting or slow hardening cement being employed; and by the mistake of using a quick-setting cement which has been re-mixed in use, and which in such a case does not again set or harden until the cement or concrete has thoroughly dried.

In both the tensile and compression tests a certain result will be obtainable before the cement can be said to have set hard, whilst in its "green" state and again at the early hours of the test, such as immediately after the final set, no hardening results are obtainable, for the test blocks will not bear any strain. In the case of the transverse tester here devised, the results are ascertainable immediately upon the final setting of the cement, and a quantitative test of the hardening properties of neat cement, and cement and sand bars, shows exactly the results that may be expected in the setting properties of the cement in use before tensile and compression tests are applicable.

It has been seen that the information given by this test, and its individual value, cannot be obtained by any other test now in vogue, such as for instance by tensile strength or compression, because in these tests no early time results, say, during the final setting and hardening of the cement, can be obtained by the apparatus.

For carrying out the transverse test special apparatus has been devised and in executing the test a useful **specification** for the preparation of test bars is as follows:—

General.—The bars shall be 6 inches long by 1 inch square. When the test-bars are gauged the same are to be marked with the trowel at the point indicated on the mould where the knife-edges of the testing machine are placed. The stress shall be applied on the rough surfaces of the bars—not on the surface which has been trowelled.

Neat Cement Bars.—For the preparation of each bar 200 grammes of cement shall be mixed with fresh water, and the temperature thereof and that of the testing-room shall be from 58° to 64° F. The quantity of water used in gauging shall be appropriate to the nature of the cement, and shall be so proportioned that the mass when gauged is of a semi-dry consistency (from 35 c.c. to 50 c.c. of water is to be used, that is, 17.5 to 25 per cent.). The cement when gauged as above shall be filled into the moulds by thumb pressure, the mould resting on an iron plate. All the cement gauged shall be pressed into the mould. When the cement has set sufficiently hard to enable the mould to be removed without injury to the bar, such removal is to be effected. The bars shall then remain in the atmosphere until required for breaking.

Sand Bars (3 plus 1).—The cement shall also be tested by means of bars prepared with three parts by weight of dry standard sand and one part cement, the said bars being of the size and shape described for neat cement tests. The mode of filling the moulds shall also be similar. The weight of dry standard sand to be used is 150 grammes, and this is to be thoroughly mixed when dry with 50 grammes of cement. The proportion of water shall be appropriate to the nature of the cement, and so proportioned that when gauged the mass is of a semi-dry consistency. From 16 c.c. to 22 c.c. water shall be used—that is, from 8 to 11 per cent. The whole of the above mixture is to be pressed into the mould, and then removed and rested in the atmosphere until tested.

NOTE.—*This Specification defines the standard quality of cement and various matters relative thereto, but does not include the provisions necessary for a contract for the sale or use of cement.*

BRITISH STANDARD SPECIFICATION FOR PORTLAND CEMENT

[REVISED 1920.]

1. Composition and Manufacture of Cement.—The cement shall be manufactured by intimately mixing together calcareous and argillaceous materials, burning them at a clinkering temperature and grinding the resulting clinker, so as to produce a cement capable of complying with this Specification.

No addition of any material shall be made after burning other than calcium sulphate, or water, or both, and then only if desired by the Vendor * and not prohibited in writing by the Purchaser.

No cement to which slag has been added or which is a mixture of Portland cement and slag will comply with this Specification.

2. Samples for Testing and by whom to be taken.—A sample or samples for testing may be taken by the Purchaser or his representative, or by any person appointed to superintend the works for the purpose of which the cement is required or his representative, or by any expert analyst employed or instructed by such Purchaser or person, or the representative of such Purchaser or person.

3. Samples for Testing and how to be taken.—Each sample for testing shall consist of approximately equal portions selected from twelve different positions in the heap or heaps when the cement is loose or from twelve different bags, barrels, or other packages, when the cement is not loose, or where there is a less number than twelve different bags, barrels, or other packages, then from each bag, barrel, or other package. Every care shall be taken in the selection, so that a fair average sample may be taken

* The term "Vendor" throughout this Specification shall mean the seller of the Cement whether he be the manufacturer of the Cement or not.

4. Sampling Large Quantities.—When more than 250 tons of cement is to be sampled at one time separate samples shall be taken, as provided in Clause 3, from each 250 tons or part thereof.

Not more than 250 tons shall be stored in such a manner that it cannot be separately identified and sampled in accordance with the provisions of this Clause and Clause 3, and separated in bulk from the remainder, *e.g.*, in the event of storage of more than 250 tons of cement in a silo, provision shall be made by which each 250 tons, or any part of 250 tons in excess thereof, shall be isolated from the remainder, and sampled at different levels.

5. Facilities for Sampling and Identifying.—The Vendor shall afford every facility, and provide all labour and materials, for taking and packing the samples for testing the cement and for subsequently identifying the cement sampled.

6. Cost of Tests, Analyses and Samples.—The tests and chemical analyses hereinafter mentioned, other than those referred to in Clause 15, shall (unless otherwise provided in the contract between the Vendor and the Purchaser) be made at the expense of the Purchaser, but no charge shall be made by the Vendor for the cement used for samples or for carriage thereon.

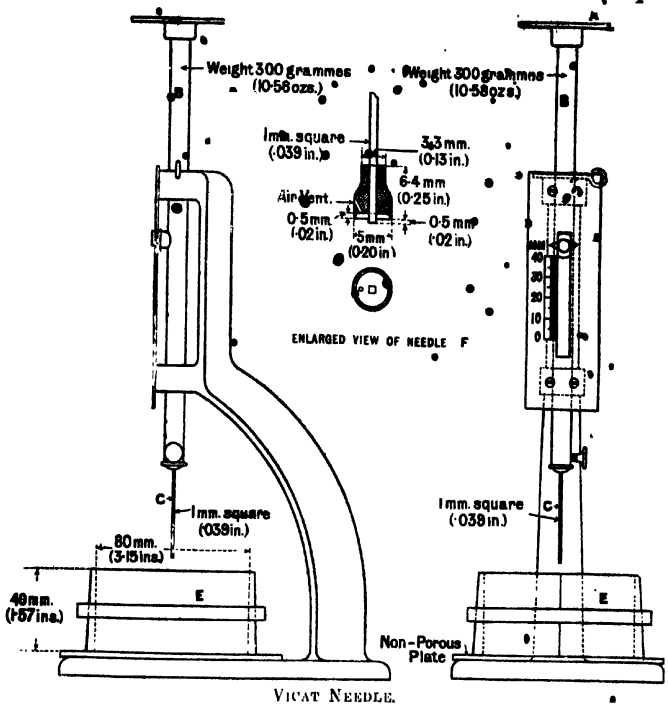
7. Tests.—The sample or samples shall be tested in the manner hereinafter mentioned for :—

(*a*) Fineness.
(*b*) Chemical composition.
(*c*) Tensile strength (neat cement).
(*d*) ,, ,, (cement and sand).
(*e*) Setting time.
(*f*) Soundness.

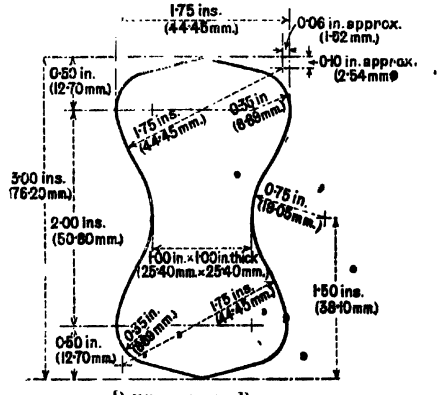
And before any sample is submitted to tests (*c*), (*d*) and (*f*), it shall be spread out for a depth of 3 inches for 24 hours in a temperature of from 58 to 64 degrees Fahrenheit.*

8. Test for Fineness.—The cement shall comply with the following conditions of fineness :—100 grammes

* The temperatures stated are applicable to temperate climates. In other climates special arrangements between Vendor and Purchaser must be made unless the temperature herein stated can be artificially obtained in the laboratory or other place where the tests are made.



VICAT NEEDLE.



DIMENSIONS OF BRIQUETTE.

(or say 4 ozs.) of cement shall be continuously sifted for a period of 15 minutes on each of the undermentioned sieves and in the order of succession given below with the following results :—

(1) The residue on a sieve $180 \times 180 = 32,400$ meshes per square inch, shall not exceed 14 per cent.

(2) The residue on a sieve $76 \times 76 = 5,776$ meshes per square inch, shall not exceed 1 per cent.

Air-set lumps in the samples may be broken down with the fingers, but nothing shall be rubbed on the sieve.

The sieves shall be prepared from wire-cloth and the diameter of the wire for the 32,400 mesh shall be .0018 inch and for the 5,776 mesh, .0044 inch. The wire-cloth shall be woven (not twilled), the cloth being carefully mounted on the frames without distortion.

9. Test for Chemical Composition.— The cement shall comply with the following conditions as to its chemical composition. The proportion of lime, after deduction of the proportion necessary to combine with the sulphuric anhydride present, to silica and alumina when calculated (in chemical equivalents) by the formula

$$\frac{CaO}{SiO_2 + Al_2O_3}$$

shall not be greater than 2.85 nor less than 2.0.* The

* EXAMPLE.—In the case of a cement containing 63.28 per cent. of lime, 21.6 per cent. of silica, 8.16 per cent. of alumina, and 2.00 per cent. of sulphuric anhydride, the proportion of lime, after deduction of the proportion necessary to combine with the sulphuric anhydride present, to silica and alumina would be as follows :—

Molecular weight of Lime ... = 56
" " " Silica ... = 60
" " " Alumina ... = 102
" " " Sulphuric Anhydride = 80

Lime combining with 2.00 per cent. of Sulphuric Anhydride $= \frac{2.00 \times 56}{80} = 1.40$ per cent.

$63.28 - 1.40 = 61.88$ per cent. Lime

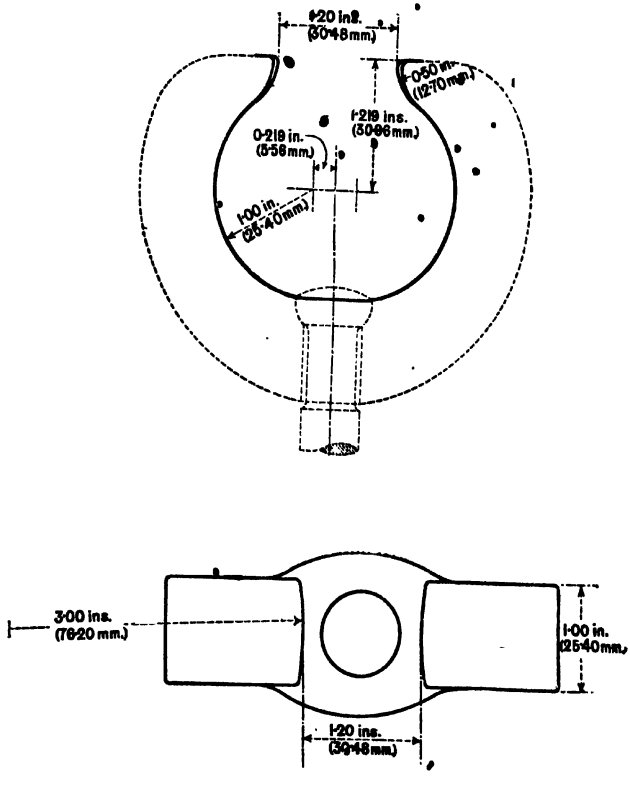
Lime $(CaO) = \frac{61.88}{56} = 1.10$

Silica $(SiO_2) = \frac{2.16}{60} = 0.36$

Alumina $(Al_2O_3) = \frac{8.16}{102} = 0.08$

Then $\frac{CaO}{SiO_2 + Al_2O_3} = \frac{1.10}{0.36 + 0.08} = 2.50$

percentage of insoluble residue shall not exceed 1.5 per cent.; that of magnesia shall not exceed 3 per cent.; and the total sulphur content calculated as sulphuric anhydride



ELEVATION AND PLAN OF JAWS FOR HOLDING BRIQUETTE.

(SO_3) shall not exceed 2.75 per cent. The total loss on ignition shall not exceed 3 per cent.

10. Test for Tensile Strength (Neat Cement).—The breaking strength of neat cement shall be ascertained from briquettes of the shape shown. The briquettes shall be prepared in the following manner :—

Preparation of Briquettes.—The cement shall be mixed with such a proportion of water that the mixture shall be plastic when filled into the moulds used for forming the briquettes.

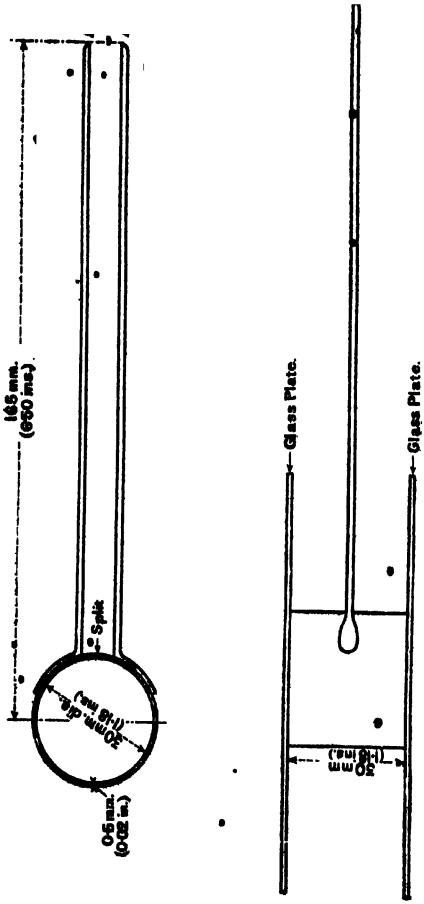
The cement, gauged as above, shall be filled into moulds of the form required to produce briquettes of the shape shown, each mould resting upon a non-porous plate. In filling the moulds the operator's hands and the blade of the ordinary gauging trowel shall alone be used. The trowel shall weigh about 7½ ozs. No ramming or hammering in any form will be permitted, nor shall any other instrument or apparatus other than the trowel before described be employed for this operation. The moulds after being filled may be shaken to the extent necessary for expelling the air.

Clean appliances shall be used for gauging, and the temperature of the water, and that of the test room at the time the above operations are being performed, shall be from 58 to to 64 degrees Fahrenheit.

The briquettes shall be kept in a damp atmosphere for 24 hours after gauging, when they shall be removed from the moulds and immediately submerged in clean fresh water and left there until taken out for breaking. The water in which they are submerged shall be renewed every seven days and shall be maintained at a temperature of between 58 and 64 degrees Fahrenheit. After they have been so taken out and until they are broken the briquettes shall not be allowed to become dry.

Breaking.—The briquettes shall be tested for breaking strength at 7 and 28 days respectively after gauging, six briquettes for each period. The breaking strength shall be the average tensile breaking strength of the six briquettes for each period. The briquettes to be tested shall be held in strong metal jaws of the shape shown, and the load shall be steadily and uniformly applied, starting from zero, and increased at the rate of 100 lbs. per square inch of section in 12 seconds.

The breaking strength of the briquettes at 7 days after



LE CHATELIER MOULD.

gauging shall be not less than 450 lbs. per square inch of section.

The breaking strength of the briquettes at 28 days after gauging shall show an increase on the breaking strength at 7 days, and shall be not less than the number of pounds per square inch of section arrived at from the following formula :—

$$\text{Breaking strength at 7 days} + \frac{40{,}000}{\text{Breaking strength at 7 days}}$$

11. Test for Tensile Strength (Cement and Sand).—The breaking strength of cement and sand shall be ascertained from briquettes also of the shape shown. The briquettes shall be prepared in the following manner :—

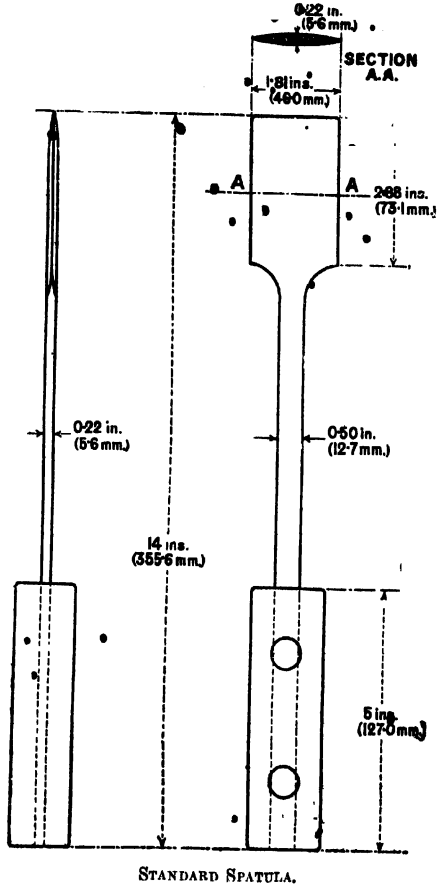
Preparation of Briquettes.—A mixture of cement and sand in the proportion of one part by weight of cement to three parts by weight of the standard sand specified on page 360 shall be gauged with sufficient water to wet the whole mass throughout without any excess of water being present.

The mixture gauged as above shall be evenly distributed in moulds of the form required to produce briquettes of the shape shown, each mould resting upon a non-porous plate. After filling a mould a small heap of the mixture shall be placed upon that in the mould and patted down with the Standard Spatula shown until the mixture is level with the top of the mould. This last operation shall be repeated a second time and the mixture patted down until water appears on the surface; the flat only of the Standard Spatula is to be used and no other instrument or apparatus is to be employed for this operation. The mould after being filled may be shaken to the extent necessary for expelling the air. No ramming or hammering in any form will be permitted during the preparation of the briquettes, which shall then be finished off in the moulds by smoothing the surface with the blade of a trowel.

Clean appliances shall be employed for gauging, and the temperature of the water and that of the test room at the time the above operations are performed shall be from 58 to 64 degrees Fahrenheit.

The briquettes shall be kept in a damp atmosphere for

24 hours after gauging, when they shall be removed from the moulds and immediately submerged in clean fresh



STANDARD SPATULA.

water, and left there until taken out for breaking. The water in which they are submerged shall be renewed

every seven days, and maintained at a temperature of between 58 and 64 degrees Fahrenheit. After they have been so taken out and until they are broken the briquettes shall not be allowed to become dry.

Breaking.—The briquettes shall be tested for breaking strength at 7 and 28 days respectively after gauging, six briquettes for each period. The breaking strength shall be the average tensile breaking strength of the six briquettes for each period. The briquettes to be tested shall be held in strong metal jaws of the shape shown, and the load steadily and uniformly applied, starting from zero, and increased at the rate of 100 lbs. per square inch of section in 12 seconds.

The breaking strength of the briquettes at 7 days after gauging shall be not less than 200 lbs. per square inch of section.

The breaking strength of the briquettes at 28 days after gauging shall show an increase on the breaking strength at 7 days and shall be not less than the number of pounds per square inch of section arrived at from the following formula :—

$$\text{Breaking strength at 7 days} + \frac{10{,}000}{\text{Breaking strength at 7 days.}}$$

Standard Sand.—The standard sand shall be obtained from Leighton Buzzard, be thoroughly washed and dried, and shall pass through a sieve of 20×20 meshes per square inch, and be retained on a sieve of 30×30 meshes per square inch. The sieves shall be prepared from wire-cloth, the wires being .0164 inch and .0108 inch in diameter respectively. The wire-cloth shall be woven (not twilled), the cloth being carefully mounted on the frames without distortion.

12. Tests for Setting Time.—Unless a specially quick setting cement is specified or required, it shall have an initial testing time of not less than 20 minutes and a final setting time of not more than 10 hours.

If a specially quick setting cement is specified or required, it shall have an initial setting time of not less than 2 minutes and a final setting time of not more than 30 minutes.

Vicat Needle Apparatus.—The initial and final setting times of the cement shall be determined by means of the Vicat needle apparatus shown.

For the purpose of carrying out the tests, a test block shall be made as follows :—

Neat cement shall be gauged in the manner and under the conditions referred to in Clause 10, and the gauging shall be completed before signs of setting occur. The test block shall then be made by filling the cement gauged as above into the Vicat mould shown at E, the mould resting upon a non-porous plate. The mould shall be completely filled, and the surface of the test block shall then be smoothed off level with the top of the mould.

Determination of Initial Setting Time.—For the determination of the initial setting time the test block confined in the mould and resting on the plate shall be placed under the rod bearing the needle; the latter shall then be lowered gently into contact with the surface of the test block and quickly released, and allowed to sink into the same.* This process shall be repeated until the needle, when brought into contact with the test block and released as above described, does not pierce it completely. The period elapsing between the time when the cement is filled into the mould and the time at which the needle ceases to pierce the test block completely shall be the initial setting time above referred to.

Determination of Final Setting Time.—For the determination of the final setting time the needle (C) of the Vicat apparatus shall be replaced by the needle (F), shown separately. The cement shall be considered as finally set when, upon applying the needle gently to the surface of the test block, the needle makes an impression thereon, while the attachment shown in the figure fails to do so. In the event of a scum forming on the surface of the test

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* The Vicat Needle may, if desired, be fitted with a mechanical attachment such as a "dash-pot," so as to ensure the steady and gentle application of the point of the needle to the surface of the test block and thereby render the test independent of the hand of the operator.

Care must be taken that the needle rests with its full weight upon the surface of the test block.

block, the underside of the test block may be used for determining the final set.

13. Test for Soundness.—The cement shall be tested for soundness by the Le Chatelier method. The apparatus for conducting the Le Chatelier test is shown. The moulds shall be kept in good condition, having the jaws not more than 0.5 mm. apart.

In conducting the test the mould shall be placed upon a small piece of glass and filled with cement gauged in the manner and under the conditions referred to in Clause 10, care being taken to keep the edges of the mould gently together whilst this operation is being performed. The mould shall then be covered with another glass plate, upon which a small weight shall be placed, and the whole shall then be immediately submerged in water at a temperature of 58 to 64 degrees Fahrenheit, and left there for 24 hours.

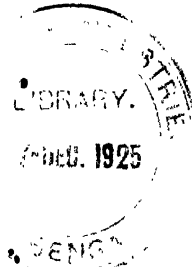
The distance separating the indicator points shall then be measured, and the mould again submerged in water at 58 to 64 degrees Fahrenheit, which shall be brought to boiling point in 25 to 30 minutes and kept boiling for six hours. The mould shall then be removed from the water and allowed to cool and the distance between the points again measured; the difference between the two measurements represents the expansion of the cement. When the sample has been aerated for 24 hours in the manner described in Clause 7, the expansion as above determined shall not exceed 10 millimetres. In the event of the cement failing to comply with this test a further test shall be made from another portion of the same sample after it shall have been aerated for a total period of 7 days in the manner before described when the expansion determined as above shall not exceed 5 mm.

14. Non-Compliance with Tests.—Any cement which does not comply with the whole of the tests and analyses hereinbefore specified or which has not been stored in the manner provided in Clause 4, may be rejected as not complying with this specification.

15. Copies of Vendor's Tests and Analyses, &c.—The vendor shall, if required, furnish free of cost a copy of any document in his possession showing the result of

any tests or analysis made for him or for any other person of any cement sold or offered for sale to the purchaser or of the lot from which the cement so sold or offered for sale has been or is to be taken, and shall, if required, at the time of purchase, furnish free of cost a certificate that the cement so sold or offered for sale has been tested and analysed, and that such tests and analysis comply in all respects with this specification, but the furnishing of such copies of documents or the giving of such certificate shall not preclude the purchaser from rejecting any cement which does not comply with this specification.

16. Delivery.—Cement shall be delivered in bags, barrels or other packages, bearing the manufacturer's name. A purchaser desiring to have the cement delivered in bags, barrels or packages sealed or of any particular size must so specify at the time of ordering.



UNITED STATES STANDARD SPECIFICATIONS AND TESTS FOR PORTLAND CEMENT.

1. DEFINITION.—Portland cement is the product obtained by finely pulverising clinker produced by calcining to incipient fusion an intimate and properly proportioned mixture of argillaceous and calcareous materials, with no additions subsequent to calcination excepting water and calcined or uncalcined gypsum.

I. Chemical Properties.

2. CHEMICAL LIMITS.—The following limits shall not be exceeded :—

Loss on ignition, per cent - - - -	4.00
Insoluble residue, per cent. - - -	0.85
Sulphuric anhydride (SO_3), per cent.	2.00
Magnesia (MgO), per cent. - - - -	5.00

II. Physical Properties.

3. SPECIFIC GRAVITY.—The specific gravity of cement shall be not less than 3.10 (3.07 for white Portland cement). Should the test of cement as received fall below this requirement a second test may be made upon an ignited sample. The specific gravity test will not be made unless specifically ordered.

4. FINENESS.—The residue on a standard No. 200 sieve shall not exceed 22 per cent. by weight.

5. SOUNDNESS.—A pat of neat cement shall remain firm and hard, and show no signs of distortion, cracking, checking, or disintegration in the steam test for soundness.

6. TIME OF SETTING.—The cement shall not develop initial set in less than 45 minutes when the Vicat needle is used or 60 minutes when the Gillmore needle is used. Final set shall be attained within 10 hours.

7. TENSILE STRENGTH.—The average tensile strength in pounds per square inch of not less than three standard mortar briquettes (see Section 50) composed of one part cement and three parts standard sand, by weight, shall be equal to or higher than the following :—

Age at Test. Days.	Storage of Briquettes	Tensile Strength. lb. per sq. in.
7	1 day in moist air, 6 days in water	200
28	1 day in moist air, 27 days in water	300

8. The average tensile strength of standard mortar at 28 days shall be higher than the strength at 7 days.

III. Packages, Marking and Storage.

9. PACKAGES AND MARKING.—The cement shall be delivered in suitable bags or barrels with the brand and name of the manufacturer plainly marked thereon, unless shipped in bulk. A bag shall contain 94 lb. net. A barrel shall contain 376 lb. net.

10. STORAGE.—The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building which will protect the cement from dampness.

IV. Inspection.

11. INSPECTION.—Every facility shall be provided the purchaser for careful sampling and inspection at either the mill or at the site of the work, as may be specified by the purchaser. At least 10 days from the time of sampling shall be allowed for the completion of the 7-day test, and at least 31 days shall be allowed for the completion of the 28-day test. The cement shall be tested in accordance with the methods hereinafter prescribed. The 28-day test shall be waived only when specifically so ordered.

V. Rejection.

12. REJECTION.—The cement may be rejected if it fails to meet any of the requirements of these specifications.

13. Cement shall not be rejected on account of failure to meet the fineness requirement if upon retest after drying at 100° C. for one hour it meets this requirement.

14. Cement failing to meet the test for soundness in steam may be accepted if it passes a retest using a new sample at any time within 28 days thereafter.

15. Packages varying more than 5 per cent. from the specified weight may be rejected; and if the average weight of packages in any shipment, as shown by weighing 50 packages taken at random, is less than that specified, the entire shipment may be rejected.

TESTS.

VI. Sampling.

16. NUMBER OF SAMPLES.—Tests may be made on individual or composite samples as may be ordered. Each test sample should weigh at least 8 lb.

17. (*a*) *Individual Sample.*—If sampled in cars one test sample shall be taken from each 50 bbl. or fraction thereof. If sampled in bins one sample shall be taken from each 100 bbl.

(*b*) *Composite Sample.*—If sampled in cars one sample shall be taken from one sack in each 40 sacks (or 1 bbl. in each 10 bbl.) and combined to form one test sample. If sampled in bins or warehouses one test sample shall represent not more than 200 bbl.

18. METHOD OF SAMPLING.—Cement may be sampled at the mill by any of the following methods that may be practicable, as ordered:—

(*a*) *From the Conveyor Delivering to the Bin.*—At least 8 lb. of cement shall be taken from approximately each 100 bbl. passing over the conveyor.

(*b*) *From Filled Bins by means of Proper Sampling Tubes.*—Tubes inserted vertically may be used for sampling

cement to a maximum depth of 10 feet. Tubes inserted horizontally may be used where the construction of the bin permits. Samples shall be taken from points well distributed over the face of the bin.

(*c*) *From Filled Bins at Points of Discharge.*—Sufficient cement shall be drawn from the discharge openings to obtain samples representative of the cement contained in the bin, as determined by the appearance at the discharge openings of indicators placed on the surface of the cement directly above these openings before drawing of the cement is started.

19. TREATMENT OF SAMPLE.—Samples preferably shall be shipped and stored in air-tight containers. Samples shall be passed through a sieve having 20 meshes per linear inch in order to thoroughly mix the sample, break up lumps and remove foreign materials.

VII. Chemical Analyses.

Loss on Ignition.

20. METHOD.—One gram of cement shall be heated in a weighed covered platinum crucible, of 20 to 25-c.c. capacity, as follows, using either method (*a*) or (*b*) as ordered:—

(*a*) The crucible shall be placed in a hole in an asbestos board; clamped horizontally so that about three-fifths of the crucible projects below, and blasted at a full red heat for 15 minutes with an inclined flame; the loss in weight shall be checked by a second blasting for 5 minutes. Care shall be taken to wipe off particles of asbestos that may adhere to the crucible when withdrawn from the hole in the board. Greater neatness and shortening of the time of heating are secured by making a hole to fit the crucible in a circular disc of sheet platinum and placing this disc over a somewhat larger hole in an asbestos board.

(*b*) The crucible shall be placed in a muffle at any temperature between 900 and 1000° C. for 15 minutes, and the loss in weight shall be checked by a second heating for 5 minutes.

21. PERMISSIBLE VARIATION.—A permissible variation of 0.25 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 4 per cent.

Insoluble Residue.

22. METHOD.—To a 1-g. sample of cement shall be added 10 c.c. of water and 5 c.c. of concentrated hydrochloric acid; the liquid shall be warmed until effervescence ceases. The solution shall be diluted to 50 c.c. and digested on a steam bath or hot plate until it is evident that decomposition of the cement is complete. The residue shall be filtered, washed with cold water, and the filter paper and contents digested in about 30 c.c. of a 5-per cent. solution of sodium carbonate, the liquid being held at a temperature just short of boiling for 15 minutes. The remaining residue shall be filtered, washed with cold water, then with a few drops of hot hydrochloric acid, 1 : 9, and finally with hot water, and then ignited at a red heat and weighed as the insoluble residue.

23. PERMISSIBLE VARIATION.—A permissible variation of 0.15 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 0.85 per cent.

Sulphuric Anhydride.

24. METHOD.—One gram of the cement shall be dissolved in 5 c.c. of concentrated hydrochloric acid diluted with 5 c.c. of water, with gentle warming; when solution is complete 40 c.c. of water shall be added, the solution filtered, and the residue washed thoroughly with water. The solution shall be diluted to 250 c.c., heated to boiling and 10 c.c. of a hot 10-per cent. solution of barium chloride shall be added slowly, drop by drop, from a pipette, and the boiling continued until the precipitate is well formed. The solution shall be digested on the steam bath until the precipitate has settled. The precipitate shall be filtered, washed, and the paper and contents placed in a weighed platinum crucible and the paper slowly charred and consumed without flaming. The barium sulphate shall then be ignited and weighed. The weight obtained multiplied

by 34.3 gives the percentage of sulphuric anhydride. The acid filtrate obtained in the determination of the insoluble residue may be used for the estimation of sulphuric anhydride instead of using a separate sample.

25. PERMISSIBLE VARIATION.—A permissible variation of 0.10 will be allowed, and all results in excess of the specified limit but within this permissible variation shall be reported as 2.00 per cent.

Magnesia.

26. METHOD.—To 0.5 g. of the cement in an evaporating dish shall be added 10 c.c. of water to prevent lumping and then 10 c.c. of concentrated hydrochloric acid. The liquid shall be gently heated and agitated until attack is complete. The solution shall then be evaporated to complete dryness on a steam or water bath. To hasten dehydration the residue may be heated to 150 or even 200° C. for one-half to one hour. The residue shall be treated with 10 c.c. of concentrated hydrochloric acid diluted with an equal amount of water. The dish shall be covered and the solution digested for ten minutes on a steam bath or water bath. The diluted solution shall be filtered and the separated silica washed thoroughly with water.* Five cubic centimetres of concentrated hydrochloric acid and sufficient bromine water to precipitate any manganese which may be present, shall be added to the filtrate (about 250 c.c.). This shall be made alkaline with ammonium hydroxide, boiled until there is but a faint odour of ammonia, and the precipitated iron and aluminium hydroxides, after settling, shall be washed with hot water, once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate shall be transferred by a jet of hot water to the precipitating vessel and dissolved in 10 c.c. of hot hydrochloric acid. The paper shall be extracted with acid, the solution and washings being added to the main solution. The aluminium and iron shall then be reprecipitated at boiling heat by ammonium hydroxide and bromine water in a volume of about 100 c.c., and the

* Since this procedure does not involve the determination of silica, a second evaporation is unnecessary.

second precipitate shall be collected and washed on the filter used in the first instance if this is still intact. To the combined filtrates from the hydroxides of iron and aluminium, reduced in volume if need be, 1 c.c. of ammonium hydroxide shall be added, the solution brought to boiling, 25 c.c. of a saturated solution of boiling ammonium oxalate added, and the boiling continued until the precipitated calcium oxalate has assumed a well-defined granular form. The precipitate after one hour shall be filtered and washed, then with the filter shall be placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner; after ignition it shall be redissolved in hydrochloric acid and the solution diluted to 100 c.c. Ammonia shall be added in slight excess, and the liquid boiled. The lime shall then be reprecipitated by ammonium oxalate, allowed to stand until settled, filtered and washed. The combined filtrates from the calcium precipitates shall be acidified with hydrochloric acid, concentrated on the steam bath to about 150 c.c., and made slightly alkaline with ammonium hydroxide, boiled and filtered (to remove a little aluminium and iron and perhaps calcium). When cool, 10 c.c. of saturated solution of sodium-ammonium-hydrogen phosphate shall be added with constant stirring. When the crystalline ammonium-magnesium orthophosphate has formed, ammonia shall be added in moderate excess. The solution shall be set aside for several hours in a cool place, filtered and washed with water containing 2.5 per cent. of NH_3. The precipitate shall be dissolved in a small quantity of hot hydrochloric acid, the solution diluted to about 100 c.c., 1 c.c. of a saturated solution of sodium-ammonium-hydrogen phosphate added, and ammonia drop by drop, with constant stirring, until the precipitate is again formed, as described and the ammonia is in moderate excess. The precipitate shall then be allowed to stand about two hours, filtered and washed as before. The paper and contents shall be placed in a weighed platinum crucible, the paper slowly charred, and the resulting carbon carefully burned off. The precipitate shall then be ignited to constant weight over a Meker burner, or a blast not strong enough to soften or melt the pyrophosphate. The weight of magnesium pyrophosphate obtained multiplied by 72.5 gives the percentage

of magnesia. The precipitate so obtained always contains some calcium and usually small quantities of iron, aluminium, and manganese as phosphates.

27. PERMISSIBLE VARIATION.—A permissible variation of 0.4 will be allowed, and all results in excess of the specified limit, but within this permissible variation shall be reported as 5.00 per cent.

VIII. Determination of Specific Gravity.

28. APPARATUS.—The determination of specific gravity shall be made with a standardized Le Chatelier apparatus which conforms to the requirements illustrated. This apparatus is standardized by the United States Bureau of Standards. Kerosene free from water, or benzine not lighter than 62° Baumé, shall be used in making this determination.

29. METHOD.—The flask shall be filled with either of these liquids to a point on the stem between zero and one cubic centimeter, and 64 g. of cement, of the same temperature as the liquid, shall be slowly introduced, taking care that the cement does not adhere to the inside of the flask above the liquid and to free the cement from air by rolling the flask in an inclined position. After all the cement is introduced, the level of the liquid will rise to some division of the graduated neck; the difference between readings is the volume displaced by 64 g. of the cement.

The specific gravity shall then be obtained from the formula—

$$\text{Specific gravity} = \frac{\text{Weight of cement (g.)}}{\text{Displaced volume (c.c.)}}$$

30. The flask, during the operation, shall be kept immersed in water, in order to avoid variations in the temperature of the liquid in the flask, which shall not exceed 0.5° C. The results of repeated tests should agree within 0.01.

31. The determination of specific gravity shall be made on the cement as received; if it falls below 3.10, a second determination shall be made after igniting the sample as described in Section 20.

IX. Determination of Fineness.

32. APPARATUS.—Wire cloth for standard sieves for cement shall be woven (not twilled) from brass, bronze, or other suitable wire, and mounted without distortion on frames not less than 1½ in. below the top of the frame. The sieve frames shall be circular, approximately 8 in. in diameter, and may be provided with a pan and cover.

33. A standard No. 200 sieve is one having nominally an 0.0029 in. opening and 200 wires per inch standardized by the U. S. Bureau of Standards, and conforming to the following requirements:—

The No. 200 sieve should have 200 wires per inch, and the number of wires in any whole inch shall not be outside the limits of 192 to 208. No opening between adjacent parallel wires shall be more than 0.0050 in. in width. The diameter of the wire should be 0.0021 in. and the average diameter shall not be outside the limits 0.0019 to 0.0023 in. The value of the sieve as determined by sieving tests made in conformity with the standard specification for these tests on a standardized cement which gives a residue of 25 to 20 per cent. on the No. 200 sieve, or on other similarly graded material, shall not show a variation of more than 1.5 per cent. above or below the standards maintained at the Bureau of Standards.

34. METHOD.—The test shall be made with 50 g. of cement. The sieve shall be thoroughly clean and dry. The cement shall be placed on the No. 200 sieve, with pan and cover attached, if desired, and shall be held in one hand in a slightly inclined position so that the sample will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the up stroke. The sieve shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall continue until not more than 0.05 g. passes through in one minute of continuous sieving. The fineness shall be determined from the weight of the residue on the sieve expressed as a percentage of the weight of the original sample.

35. Mechanical sieving devices may be used, but the cement shall not be rejected if it meets the fineness re-

quirement when tested by the hand method described in Section 34.

A. Mixing Cement Pastes and Mortars.

36. METHOD.—The quantity of dry material to be mixed at one time shall not exceed 1000 g. nor be less than 500 g. The proportions of cement or cement and sand shall be stated by weight in grams of the dry materials; the quantity of water shall be expressed in cubic centimetres (1 c.c. of water = 1 g.). The dry materials shall be weighed, placed upon a non-absorbent surface, thoroughly mixed dry if sand is used, and a crater formed in the centre, into which the proper percentage of clean water shall be poured; the material on the outer edge shall be turned into the crater by the aid of a trowel. After an interval of half a minute for the absorption of the water the operation shall be completed by continuous, vigorous mixing, squeezing and kneading with the hands for at least one minute.* During the operation of mixing, the hands should be protected by rubber gloves.

37. The temperature of the room and the mixing water shall be maintained as nearly as practicable at 21° C. (70° F.).

XI. Normal Consistency.

38. APPARATUS.—The Vicat apparatus consists of a frame *A* bearing a movable rod *B*, weighing 300 g., one end *C* being 1 cm. in diameter for a distance of 6 cm., the other having a removable needle *D*, 1 mm. in diameter, 6 cm. long. The rod is reversible, and can be held in any

* In order to secure uniformity in the results of tests for the time of setting and tensile strength the manner of mixing above described should be carefully followed. At least one minute is necessary to obtain the desired plasticity which is not appreciably affected by continuing the mixing for several minutes. The exact time necessary is dependent upon the personal equation of the operator. The error in mixing should be on the side of over mixing.

desired position by a screw E, and has midway between the ends a mark F which moves under a scale (graduated to millimetres) attached to the frame A. The paste is held in a conical, hard-rubber ring G, 7 cm. in diameter at the base, 4 cm. high, resting on a glass plate H about 10 cm. square.

39. METHOD.—In making the determination, 500 g. of cement, with a measured quantity of water, shall be kneaded into a paste, as described in Section 36, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained about 6 in. apart; the ball resting in the palm of one hand shall be pressed into the larger end of the rubber ring held in the other hand, completely filling the ring with paste; the excess at the larger end shall then be removed by a single movement of the palm of the hand; the ring shall then be placed on its larger end on a glass plate and the excess paste at the smaller end sliced off at the top of the ring by a single oblique stroke of a trowel held at a slight angle with the top of the ring. During these operations care shall be taken not to compress the paste. The paste confined in the ring, resting on the plate, shall be placed under the rod, the larger end of which shall be brought in contact with the surface of the paste; the scale shall be then read, and the rod quickly released. The paste shall be of normal consistency when the rod settles to a point 10 mm. below the original surface in half a minute after being released. The apparatus shall be free from all vibrations during the test. Trial pastes shall be made with varying percentages of water until the normal consistency is obtained. The amount of water required shall be expressed in percentage by weight of the dry cement.

40. The consistency of standard mortar shall depend on the amount of water required to produce a paste of normal consistency from the same sample of cement. Having determined the normal consistency of the sample, the consistency of standard mortar made from the same sample shall be as indicated in Table I., the values being in percentage of the combined dry weights of the cement and standard sand.

TABLE I.—PERCENTAGE OF WATER FOR STANDARD MORTARS.

Percentage of Water for Neat Cement Paste of Normal Consistency	Percentage of Water for One Cement, Three Standard Ottawa Sand	Percentage of Water for Neat Cement Paste of Normal Consistency	Percentage of Water for One Cement, Three Standard Ottawa Sand
15	9.0	23	10.3
16	9.2	24	10.5
17	9.3	25	10.7
18	9.5	26	10.8
19	9.7	27	11.0
20	9.8	28	11.2
21	10.0	29	11.3
22	10.2	30	11.5

XII. Determination of Soundness.*

41. APPARATUS.—A steam apparatus, which can be maintained at a temperature between 98 and 100° C., or one similar to that shown is recommended. The capacity of this apparatus may be increased by using a rack for holding the pats in a vertical or inclined position.

42. METHOD.—A pat from cement paste of normal consistency about 3 in. in diameter, $\frac{1}{2}$ in. thick at the centre, and tapering to a thin edge, shall be made on clean glass plates about 4 in. square, and stored in moist air for 24 hours. In moulding the pat, the cement paste shall first

* Unsoundness is usually manifested by change in volume which causes distortion, cracking, checking or disintegration.

Pats improperly made or exposed to drying may develop what are known as shrinkage cracks within the first 24 hours and are not an indication of unsoundness.

The failure of the pats to remain on the glass or the cracking of the glass to which the pats are attached does not necessarily indicate unsoundness.

be flattened on the glass and the pat then formed by drawing the trowel from the outer edge toward the centre.

43. The pat shall then be placed in an atmosphere of steam at the temperature between 98° and 100° C. upon a suitable support 1 in. above boiling water for 5 hours.

44. Should the pat leave the plate, distortion may be detected best with a straight edge applied to the surface which was in contact with the plate.

XIII. Determination of Time of Setting.

45. The following are alternate methods, either of which may be used as ordered :—

46. VICAT APPARATUS.—The time of setting shall be determined with the Vicat apparatus described in Section 38.

47. VICAT METHOD.—A paste of normal consistency shall be moulded in the hard-rubber ring *G* as described in Section 39, and placed under the rod *B*, the smaller end of which shall then be carefully brought in contact with the surface of the paste, and the rod quickly released. The initial set shall be said to have occurred when the needle ceases to pass a point 5 mm. above the glass plate in half a minute after being released; and the final set, when the needle does not sink visibly into the paste. The test pieces shall be kept in moist air during the test. This may be accomplished by placing them on a rack over water contained in a pan and covered by a damp cloth, kept from contact with them by means of a wire screen; or they may be stored in a moist closet. Care shall be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point may increase the penetration. The time of setting is affected not only by the percentage and temperature of the water used and the amount of kneading the paste receives, but by the temperature and humidity of the air, and its determination is therefore only approximate.

48. GILLMORE NEEDLES.—The time of setting shall be determined by the Gillmore needles. The Gillmore needles should preferably be mounted.

49. GILLMORE METHOD.—The time of setting shall be

determined as follows:—A pat of neat cement paste about 3 in. in diameter and $\frac{1}{2}$ in. in thickness with a flat top mixed to a normal consistency, shall be kept in moist air at a temperature maintained as nearly as practicable at 21° C. (70° F.). The cement shall be considered to have acquired its initial set when the pat will bear, without appreciable indentation, the Gillmore needle $\frac{1}{12}$ in in diameter, loaded to weigh $\frac{1}{4}$ lb. The final set has been acquired when the pat will bear without appreciable indentation the Gillmore needle $\frac{1}{24}$ in. in diameter, loaded to weigh 1 lb. In making the test, the needles shall be held in a vertical position, and applied lightly to the surface of the pat.

XIV. Tension Tests.

50. Form of Test Piece. The form of test piece shown shall be used. The moulds shall be made of non-corroding metal and have sufficient material in the sides to prevent spreading during moulding. Gang moulds when used shall be of the type shown. Moulds shall be wiped with an oily cloth before using.

51. Standard Sand.—The sand to be used shall be natural sand from Ottawa, Ill., screened to pass a No. 20 sieve, and retained on a No. 30 sieve. This sand may be obtained from the Ottawa Silica Co., at a cost of three cents per pound, f. o. b. cars, Ottawa, Ill.

52. This sand, having passed the No. 20 sieve, shall be considered standard when not more than 5 g. pass the No. 30 sieve after one minute continuous sieving of a 500-g. sample.

53. The sieves shall conform to the following specifications :—

The No. 20 sieve shall have between 19.5 and 20.5 wires per whole inch of the warp wires and between 19 and 21 wires per whole inch of the shoot wires. The diameter of the wire should be 0.0165 in. and the average diameter shall not be outside the limits of 0.0160 and 0.0170 in.

The No. 30 sieve shall have between 29.5 and 30.5 wires per whole inch of the warp wires and between 28.5 and 31.5 wires per whole inch of the shoot wires. The diameter of the wire should be 0.0110 in. and the average diameter shall not be outside the limits 0.0105 to 0.0115 in.

54. MOULDING.—Immediately after mixing, the standard mortar shall be placed in the moulds, pressed in firmly with the thumbs and smoothed off with a trowel without ramming. Additional mortar shall be heaped above the mould and smoothed off with a trowel; the trowel shall be drawn over the mould in such a manner as to exert a moderate pressure on the material. The mould shall then be turned over and the operation of heaping, thumbing and smoothing off repeated.

55. TESTING.—Tests shall be made with any standard machine. The briquettes shall be tested as soon as they are removed from the water. The bearing surfaces of the clips and briquettes shall be free from grains of sand or dirt. The briquettes shall be carefully centred and the load applied continuously at the rate of 600 lb. per minute.

56. Testing machines should be frequently calibrated in order to determine their accuracy.

57. FAULTY BRIQUETTES.—Briquettes that are manifestly faulty, or which give strengths differing more than 15 per cent. from the average value of all test pieces made from the same sample and broken at the same period, shall not be considered in determining the tensile strength.

XV. Storage of Test Pieces.

58. APPARATUS.—The moist closet may consist of a soapstone, slate, or concrete box, or a wooden box lined with metal. If a wooden box is used, the interior should be covered with felt or broad wicking kept wet. The bottom of the moist closet should be covered with water. The interior of the closet should be provided with non-absorbent shelves on which to place the test pieces, the shelves being so arranged that they may be withdrawn readily.

59. METHODS.—Unless otherwise specified, all test pieces, immediately after moulding, shall be placed in the moist closet for from 20 to 24 hours.

60. The briquettes shall be kept in moulds on glass plates in the moist closet for at least 20 hours. After 24 hours in moist air the briquettes shall be immersed in clean water in storage tanks of non-corroding material.

61. The air and water shall be maintained as nearly as practicable at a temperature of 21°C. (70° F.).

FRENCH GOVERNMENT SPECIFICATION FOR PORTLAND CEMENT.

MINISTRY OF PUBLIC WORKS, BRIDGES AND HIGHWAYS.

MINISTERIAL DECREE RELATING TO SUPPLIES OF CEMENT AND HYDRAULIC LIME.

GENERAL CONDITIONS.

ARTICLE 1.—All supplies of cement and lime used upon the works of the Administration of Public Works shall comply with the following requirements so far as they may apply, whether the materials are supplied direct to the Administration or to Contractors for use upon contracts entered into with the Administration.

PART I.

Clauses Applicable to all Contracts. Mode of Delivery.

ARTICLE 2.—The cement shall be delivered in sacks or in casks.

Sacks shall contain a nett weight of 50 kilogrammes (110 lbs.). They must be stitched inside and sealed by means of a lead seal bearing the mark of the maker, and of a design accepted by the Administration.

The casks shall bear on one of the heads the brand of the maker, and on the other the nett weight of cement contained therein.

The sacks and casks must be in good condition at the time of delivery.

All wet cement such as has been spoiled by damp will be rejected.

A Bill of Lading or delivery note must be forwarded to the Engineer in respect of each and every consignment.

Storage.

ARTICLE 3.—The sacks or casks of cement shall be kept in dry stores, closed and roofed. Each consignment must be kept separate and distinct.

The contractor shall be responsible for the keeping of cement in store up to the time of using, except as stipulated in Article 17.

All sacks or casks of cement or lime which are found damaged or whose coverings are not in good condition at the time of delivery will be rejected.

Tests.

ARTICLE 4.—No cement may be used before having been submitted to the tests prescribed by the special schedule of instructions of the contract and thus received provisionally.

The Engineer will have the right to re-test during the whole time that the cement lies provisionally in the store, and to reject any consignment which may fail on re-testing to pass the specification.

In the event of the tests having given unfavourable results, the Contractor may request that such tests be again carried out at the Laboratory of the Ecole des Ponts et Chaussées (School of Bridges and Highways).

Sampling.

ARTICLE 5.—The samples of cement to be submitted for testing shall be taken from such different points of several sacks, casks, or bins, as may be indicated by the Engineer. The cement coming from different consignments must not be mixed.

Quality.

ARTICLE 6.—The cement must be of uniform composition and quality. It must not contain either under-burnt material or any adulterating ingredient.

Fineness of Grinding.

ARTICLE 7.—The tests shall be carried out on a sample of 100 grammes (about 3½ ounces).

The sieving will be effected by means of sieves of 324, 900 or 4,900 meshes per square centimetre. The wire of the 3 sieves will be of the respective sizes of 20, 15 or 5 hundredths of millimetres.

Apparent Density. (Weight per Bushel.)

ARTICLE 8.—The weight will be determined by pouring the cement gently without pressure into a cylindrical metal measure, having a capacity of 1 litre per 10 centimetres depth.

The cement contained in the measure will be weighed, and the apparent density will be taken as the average of 3 successive weighings.

In case of dispute the test may be retaken with the aid of a hopper with sieves with holes of 2 millimetres. This hopper will be placed so that the end of its lower shoot is 5 centimetres above the measure.

The cement or lime must be poured into the measure in such a way as to avoid all shock or shaking.

When the measure is overflowing the surplus must be taken off by drawing over the top a straight-edge held vertically.

Setting.

ARTICLE 9.—The cement shall be gauged with fresh water to a firm paste and placed in the form of pats of about 4 centimetres thickness and these shall be immediately immersed in fresh water or in sea water as may be prescribed by the special schedule of instructions under the contract.

The cement, the water for gauging, and the bath for immersion must be of a temperature of at least 15° centigrade when it is a question of determining the initial set, and at the most 15° when it is a question of finding the time of the final set.

The initial set shall be taken to be at the moment when a Vicat needle having a section of 1 millimetre square, and weighing 300 grammes, no longer pierces through the pat.

The final set will be taken to be when the surface of the pat supports the same needle without it penetrating any appreciable distance, such as a tenth of a millimetre.

In case of dispute it will be considered that the paste of cement is firm which when gauged at the rate of 5 minutes per kilogramme, and then placed in a box 4 centimetres deep, may be pierced to the depth of 6 millimetres from the bottom of the box by a rod of the diameter of 1 centimetre and of the weight of 300 grammes.

Tensile Strain.

ARTICLE 10.—The tests for strength shall be carried out with a firm paste of neat cement and with a plastic mortar of cement gauged with fresh water. They will be made by means of briquettes in the form of an 8, having a section in the middle of 5 square centimetres.

The moulds for making the briquettes shall all be filled at one time, and when filled must be shaken in order to expel the air bubbles; the paste or the mortar should then be pressed with a trowel, but not hammered. Then with the blade of a trowel the surplus should be taken from the top of the moulds and the surface smoothed.

Each test will consist of the breaking of these briquettes, and the tensile strength will be taken as the average of the 4 highest results.

The mortar shall be mixed by weight in the proportion of 1 part of cement and 3 of dry sand. The sand shall be composed of equal parts of grains of 3 sizes separated by 4 sieves perforated with holes of $\frac{1}{2}$, 1 and 2 millimetres diameter.

The briquettes having been kept in a damp atmosphere and shaded from draughts and from the sun for the length of time fixed by the special schedule of instructions under the contract, shall be taken out of the moulds and immersed in fresh water or sea water as may have been prescribed by the schedule. In any case the water will be renewed every 7 days.

In the event of dispute the firm paste of neat cement shall be taken as defined in article 9, and the plastic mortar shall be the mortar made with sand from the beach of Leucante, supplied by the Administration, and gauged with a quantity of water in the proportions of 1 kilogramme of cement and sand to 50 grammes, 1-5th pp. being the weight of water necessary to transform a kilogramme of cement into a firm paste.

Contraction or Expansion in Heat or Cold.

ARTICLE 11.—The tests for contraction in cold will be made with pats of cement gauged with fresh water to a firm paste. The pats being about 10 centimetres diameter (about 4 inches) and 2 centimetres thickness (about ¾ an inch) will be thinned off at the edges and placed on squares of glass. The pats will be immersed (under the conditions fixed by the special schedule of instructions under the contract), and kept in water until the final acceptance of the cement.

None of the pats must show the slightest trace of swelling, cracking or blowing. The edges of the pats must remain attached to the glass and not lift at any point.

The tests for expansion in heat will be made on cylindrical briquettes of a diameter and length of 30 millimetres, moulded in a tin tube ½ millimetre thick, split down one side, and having soldered on each side of the slit a needle of 150 millimetres in length.

Twenty-four hours after the final set these briquettes will be immersed in water which will be gradually increased to the temperature fixed by the schedule of instructions, and maintained at this temperature during the time, likewise fixed by the schedule, and then allowed to cool down to the initial temperature.

The increase in the distance between the positions of the needles must not exceed that indicated in the special schedule of instructions.

Constancy of Temperature.

ARTICLE 12.—The water in which the briquettes and pats are kept must be maintained at a temperature between 12° and 18° Centigrade, 53.6° F.—64.4° F.

Removal of Rejected Cement.

ARTICLE 13.—The rejected cement must be taken away from the store at the risk and expense of the Contractor within 10 days dating from the notification of the rejection.

In the event of the Contractor not conforming to this rule the Engineer will attend to the removal of the rejected cement. It will be taken away and deposited at the expense, risk and responsibility of the Contractor in a store hired on his account.

PART II.

Clauses Applicable to Contracts for Supplies Without Labour.

Orders.

ARTICLE 14.—The cement will be supplied in successive lots under penalties for delays in delivery which will be stated on the requisitions.

The special schedule of instructions under the contract will determine the maximum quantity of cement which may be required monthly from the contractor, as well as the minimum time allowed for the execution of each order.

In case of delay in delivery the Administration may, 10 days after notification to the Contractor by the Engineer, purchase at his risk and expense the quantity of cement which shall not have been delivered.

If the cement supplied gives cause for rejection, and if the Contractor being in arrears has not replaced the rejected cement within 10 days or such longer time as may be fixed by the Engineer, the Administration may likewise buy at the expense of the Contractor a quantity of cement equal to that which may have been rejected.

In both cases the amount of the contract will be diminished by the quantity purchased through the default of the Contractor, and the Contractor shall not be able to claim the benefit of Article 31 of the general clauses and conditions.

Return of Packing.

ARTICLE 15.—The empty sacks will be returned to the store by the Administration as soon as may be convenient and held at the disposal of the Contractor, who must remove them at his own expense.

In default of removal within the time fixed by the Engineer, and in case of incumbering the store, the empty sacks will be returned immediately to the Contractor at his address and at his expense.

The value of sacks not returned will be paid to the Contractor at the end of the contract at the price fixed by the schedule of contract conditions without rebate.

The empty casks will remain the property of the Government.

Weight.

ARTICLE 16.—All the sacks or casks will be weighed by way of check at the time of arrival at the store.

The tare will be determined by weighing a number of sacks or casks equal to about 10 per cent. of the total number.

Receipt.

ARTICLE 17.—The provisional receipt of each lot of supplies will be acknowledged as soon as the cement composing the consignment shall have passed the tests fixed by the schedule of contract conditions.

The cement which shall not have been used within six months from receipt shall be counted as accepted at the expiration of this period after it satisfies all the prescribed conditions, and the contractor shall from that moment be discharged of all responsibility for its keeping and storage.

Certificates.

ARTICLE 18.—Certificates will be issued within one year dating from the provisional receipt of the last consignment.

SCHEDULE OF INSTRUCTIONS FOR CEMENT TO BE USED ON SEA WORKS.

Description of Product.

ARTICLE 1.—The Portland cement must be produced by the grinding of an intimate mixture of carbonate of lime, silica, alumina, and ferric oxide burnt to the point of incipient fusion.

Name and Supervision of Works.

ARTICLE 2.—The cement must come exclusively and direct from the works.

The Administration reserves to itself the right to examine the whole process of manufacture, storage, and despatch of the cement to be supplied in execution of the present contract, and may appoint a permanent representative at the works for the purpose of this inspection.

Method of Delivery.

ARTICLE 3.—The cement shall be delivered free (in sacks or casks).

Chemical Composition.

ARTICLE 4.—The cement must not contain more than 1.5 per cent. of sulphuric anhydride, nor more than 2 per cent. of magnesia, nor more than 8 per cent. of alumina, nor sulphides in appreciable quantities.

Its hydraulic index, that is to say, the relation between the weight of the combined silica and alumina of the one part, and the weight of lime and magnesia of the other part will be at least .47 to 8 per cent. of alumina, with a diminution of .02 for every 1 per cent. of alumina below 8 per cent.

Fineness of Grinding.

ARTICLE 5.—The cement must leave not more than 10 per cent. of its weight on a sieve with 4,900 meshes per square centimetre, and at the most 2 per cent. on a sieve of 324 meshes.

Apparent Density.

ARTICLE 6.—The weight of a litre of cement will be 1,200 grammes (96 lbs. per bushel), at least.

Time of Setting.

ARTICLE 7.—The cement plunged into the fresh water must not begin to set before 20 minutes.

Tensile Strain of Neat Cement.

ARTICLE 8.—The briquettes of neat cement plunged into sea water at the end of 24 hours shall show the tensile strain per square centimetre of at least 15 kilogrammes (212 lbs. per square inch), in 7 days, and 30 kilogrammes (425 lbs. per square inch), in 28 days. The strain must, moreover, increase by at least 3 kilogrammes (43 lbs.) from the 7th to the 28th day.

NOTE.—*These figures are minimum. The Engineers can increase them after having assured themselves that the works can supply what is specified.*

PORTLAND CEMENT.

USEFUL TABLES.

METRICAL EQUIVALENTS.

1	Millimetre	=	.039	inch	1	Litre	= 1.0 kilogramme
1	Metre	=	39.37	inches	1	Litre	= 61.0 cubic inches
1	Kilometre	=	3280.9	feet	1	Litre	= .035 cubic foot
1	Sq. centimetre	=	.155	sq. inch	1	Kilogramme	= 2.2 lbs.
1	Square metre	=	10.76	square feet	1	Kilogramme per square c/m	= 14.22 lbs. per sq. inch
1	Cubic centimetre	=	.061	cubic inch	1	Kilogramme per square metre	= .205 lbs. per sq. foot
1	Cubic metre	=	35.3	cubic feet	1	Calorie per kilogramme	= 1.8 B.T.U. per lb.
1	Litre	=	1.76	pints			
1	Litre	=	.22	gallons			

1	Inch	=	25.4	millimetres	1	Cubic foot of water	= .028 ton
1	Foot	=	.305	metre	1	Cubic foot of water	= 62.43 lbs. at 39.1° F.
1	Yard	=	.914	metre	1	Cubic foot of water	= 62.35 lbs. at 62° F.
1	Square inch	=	6.451	square centimetres	1	Foothead	= .434 lbs. per sq. in.
1	Square foot	=	.093	square metre	1	Pound of water	= 27.72 cubic inches
1	Square yard	=	.836	square metre	1	,, ,,	= .10 gallon
1	Cubic inch	=	16.386	cubic centimetres	1	Cwt. of water	= 11.2 gallons
1	Cubic foot	=	.028	cubic metre	1	,, ,,	= 1.80 cubic feet
1	Cubic yard	=	.764	cubic metre	1	Ton ,,	= 35.97 cubic feet
1	Pound (16 oz.)	=	.454	kilogramme	1	,, ,,	= 224 gallons
1	Cwt. (112 lbs.)	=	50.80	kilogrammes	1	,, ,,	= 1,000 litres [(approx.).
1	Ton (20 cwts.)	=	1,016	kilogrammes	1	,, ,,	= 1 cubic metre [(approx).
1	Gallon	=	10	lbs.	1	Cubic metre of water	= 220 gallons
1	,,	=	277.27	cubic inches	1	Cubic metre of water	= 1.31 cubic yards
1	,,	=	.16	cubic foot	1	Cubic metre of water	= 61,028 cubic inches
1	,,	=	4.54	litres	1	Cubic metre of water	= 35.32 cubic feet
1	Cubic inch of water	=	.036	lbs.	1	Cubic metre of water	= 1,000 kilos (at 4° C.)
1	Cubic inch of water	=	.0036	gallons	1	Cubic metre of water	= 1 ton (approx.)
1	Cubic foot of water	=	6.24	gallons	1	Cubic metre of water	= 1,000 litres
1	Cubic foot of water	=	28.32	litres			
1	Cubic foot of water	=	.028	cubic metre			
1	Cubic foot of water	=	.557	cwt.			

TO CONVERT—				
Lbs. per square inch	×	.07	=	Kilos per square c/m.
Lbs. per square foot	×	4.88	=	Kilos per square metre
Kilos per square c/m.	×	14.2	=	Lbs. per square inch
Kilos per square metre	×	0.2	=	Lbs. per square foot
Grains per gallon	×	0.014	=	Grammes per litre
Grammes per litre	×	73.09	=	Grains per gallon
Gallons per square foot	×	48.91	=	Litres per square metre
Litres per square metre	×	0.02	=	Gallons per square foot
Heat units	×	0.252	=	Calories
Calories	×	3.968	=	Heat units

English Weights and Metrical Equivalents.

Cwts.	Kilos.	Cwts.	Kilos.	Tons.	Kilos.	Tons.	Kilos.	Tons	Kilos.
1	51	11	559	1	1016	12	12193	35	35562
2	102	12	610	2	2032	13	13209	40	40642
3	152	13	660	3	3048	14	14225	45	45722
4	203	14	711	4	4064	15	15241	50	50802
5	254	15	762	5	5080	16	16257	55	55883
6	305	16	813	6	6095	17	17273	60	60963
7	356	17	864	7	7112	18	18289	65	66043
8	406	18	914	8	8128	19	19305	70	71123
9	457	19	965	9	9144	20	20321	80	81284
10	508	—	—	10	10160	25	25401	90	91444
—	—	—	—	11	11176	30	30481	100	101605

Weights of Building Materials.

A bushel of chalk lime (dry) weighs 50 lbs., and a bushel of stone lime (dry), 56 lbs. A measure is 1 cub. yd., and contains eighteen heaped bushels, or twenty-three striked bushels; from this the weights can be calculated. A bushel of Portland cement (dry) weighs from 105 lb. to 110 lb. A bushel of hair for plaster, 14 lbs. to 15 lbs. A load of 500 bricks, 9 in. by 4½ in. by 2¾ in., weighs about 32½ cwt., and a load of 1,000 plain tiles, 10½ in. by 6½ in. by ⅝ in., weighs about 23 cwt.; a load or cubic yard of sand, 26 cwt. to 28½ cwt.; the same quantity of building mortar mixed (semi-dry), 24 cwt. to 26 cwt. Approximately, as filled into carts, 21 cub. ft. of river sand weigh a ton; pit sand requires 22 cub. ft. to weigh a ton, under the same conditions.

21	cub. ft.	of river sand	=	1	ton approximately.
22	,,	pit sand	=	,,	,,
22	,,	Thames ballast	=	,,	,,
21	,,	gravel	=	,,	,,
60	,,	ashes	=	,,	,,
25½	,,	shingle	=	,,	,,

The weight of sand as given by different authorities ranges as follows:—Pit sand, 90 lb. to 100 lb. per cubic foot; Thames, 91 lb. to 102 lb.; river sand, 117 lb. to 118 lb.

DECIMAL FRACTIONS OF A LINEAL INCH IN MILLIMETRES.

Inch	Milli-metres	Inch	Milli-metres	Inch	Milli-metres
.01	.254	.39	9.906	.77	19.558
.02	.508	.40	10.160	.78	19.812
.03	.762	.41	10.414	.79	20.066
.04	1.016	.42	10.668	.80	20.320
.05	1.270	.43	10.922	.81	20.574
.06	1.254	.44	11.176	.82	20.828
.07	1.778	.45	11.430	.83	21.082
.08	2.032	.46	11.684	.84	21.336
.09	2.286	.47	11.938	.85	21.590
.10	2.540	.48	12.192	.86	21.844
.11	2.794	.49	12.446	.87	22.098
.12	3.048	.50	12.700	.88	22.352
.13	3.302	.51	12.954	.89	22.606
.14	3.556	.52	13.208	.90	22.860
.15	3.810	.53	13.462	.91	23.114
.16	4.064	.54	13.716	.92	23.368
.17	4.318	.55	13.970	.93	23.622
.18	4.572	.56	14.224	.94	23.876
.19	4.826	.57	14.478	.95	24.130
.20	5.080	.58	14.732	.96	24.384
.21	5.334	.59	14.986	.97	24.638
.22	5.588	.60	15.240	.98	24.892
.23	5.842	.61	15.494	.99	25.146
.24	6.096	.62	15.748	1.00	25.400
.25	6.350	.63	16.002	2.00	50.799
.26	6.604	.64	16.256	3.00	76.199
.27	6.858	.65	16.510	4.00	101.598
.28	7.112	.66	16.764	5.00	126.998
.29	7.366	.67	17.018	6.00	152.397
.30	7.620	.68	17.272	7.00	177.797
.31	7.874	.69	17.526	8.00	203.196
.32	8.128	.70	17.780	9.00	228.596
.33	8.382	.71	18.034	10.00	253.995
.34	8.636	.72	18.288	11.00	279.395
.35	8.890	.73	18.542	12.00	304.794
.36	9.114	.74	18.796	= 1 foot	
.37	9.398	.75	19.050	—	—
.38	9.652	.76	19.304	—	—

METRES IN LINEAL YARDS.

Metres	Yards	Metres	Yards	Metres	Yards
1	1.094	35	38.277	68	74.366
2	2.188	36	39.370	69	75.460
3	3.281	37	40.464	70	76.553
4	4.374	38	41.558	71	77.647
5	5.468	39	42.651	72	78.741
6	6.562	40	43.745	73	79.834
7	7.655	41	44.838	74	80.928
8	8.749	42	45.932	75	82.021
9	9.843	43	47.026	76	83.115
10	10.936	44	48.119	77	84.209
11	12.030	45	49.213	78	85.302
12	13.123	46	50.306	79	86.396
13	14.217	47	51.400	80	87.490
14	15.311	48	52.494	81	88.583
15	16.404	49	53.587	82	89.677
16	17.498	50	54.681	83	90.770
17	18.591	51	55.775	84	91.864
18	19.685	52	56.868	85	92.958
19	20.779	53	57.962	86	94.051
20	21.872	54	59.055	87	95.145
21	23.966	55	60.149	88	96.239
22	24.060	56	61.243	89	97.332
23	25.153	57	62.336	90	98.426
24	26.247	58	63.430	91	99.519
25	27.340	59	64.524	92	100.613
26	28.434	60	65.617	93	101.707
27	29.528	61	66.711	94	102.800
28	30.621	62	67.804	95	103.894
29	31.715	63	68.898	96	104.987
30	32.809	64	69.992	97	106.081
31	33.902	65	71.085	98	107.175
32	34.996	66	72.179	99	108.268
33	36.089	67	73.272	100	109.362
34	37.183	—	—	—	—

Kilogrammes in Pounds.

Kilos	Pounds	Kilos	Pounds	Kilos	Pounds
1	2.205	35	77.162	68	149.914
2	4.409	36	79.366	69	152.119
3	6.614	37	81.571	70	154.323
4	8.818	38	83.776	71	156.528
5	11.023	39	85.980	72	158.733
6	13.228	40	88.185	73	160.937
7	15.432	41	90.389	74	163.142
8	17.637	42	92.594	75	165.347
9	19.842	43	94.799	76	167.551
10	22.046	44	97.003	77	169.756
11	24.251	45	99.208	78	171.960
12	26.455	46	101.413	79	174.165
13	28.660	47	103.617	80	176.370
14	30.865	48	105.822	81	178.574
15	33.069	49	108.026	82	180.779
16	35.274	50	110.231	83	182.983
17	37.479	51	112.436	84	185.118
18	39.683	52	114.640	85	187.393
19	41.888	53	116.845	86	189.597
20	44.092	54	119.049	87	191.802
21	46.297	55	121.254	88	194.010
22	48.502	56	123.459	89	196.211
23	50.706	57	125.663	90	198.416
24	52.911	58	127.868	91	200.620
25	55.115	59	130.073	92	202.825
26	57.320	60	132.277	93	205.030
27	59.525	61	134.482	94	207.234
28	61.729	62	136.486	95	209.439
29	63.934	63	138.891	96	211.644
30	66.139	64	141.096	97	213.848
31	68.343	65	143.300	98	216.053
32	70.548	66	145.505	99	218.275
33	72.752	67	147.710	100	220.462
34	74.957	—	—	—	—

Pounds per Sq. In. in Kilograms per Sq. Cent.

Pounds per sq. inch	Kilograms per square centimetre	Pounds per sq. inch	Kilograms per square centimetre	Pounds per sq. inch	Kilograms per square centimetre	Pounds per sq. inch	Kilograms per square centimetre
1	0.0703	41	2.8826	81	5.6948	205	14.4129
2	0.1406	42	2.9529	82	5.7652	210	14.7645
3	0.2109	43	3.0232	83	5.8355	215	15.1160
4	0.2812	44	3.0935	84	5.9058	220	15.4675
5	0.3515	45	3.1638	85	5.9761	225	15.8191
6	0.4218	46	3.2341	86	6.0464	230	16.1706
7	0.4921	47	3.3044	87	6.1167	235	16.5221
8	0.5624	48	3.3747	88	6.1870	240	16.8737
9	0.6328	49	3.4450	89	6.2573	245	17.2252
10	0.7031	50	3.5153	90	6.3276	250	17.5767
11	0.7734	51	3.5856	91	6.3979	255	17.9283
12	0.8437	52	3.6559	92	6.4682	260	18.2798
13	0.9140	53	3.7263	93	6.5385	265	18.6313
14	0.9843	54	3.7966	94	6.6088	270	18.9829
15	1.0546	55	3.8669	95	6.6722	275	19.3344
16	1.1249	56	3.9372	96	6.7495	280	19.6860
17	1.1952	57	4.0075	97	6.8198	285	20.0375
18	1.2655	58	4.0778	98	6.8901	290	20.3890
19	1.3358	59	4.1481	99	6.9604	295	20.7406
20	1.4062	60	4.2184	100	7.0307	300	21.0921
21	1.4764	61	4.2887	105	7.3822	310	21.7951
22	1.5467	62	4.3590	110	7.7338	320	22.4981
23	1.6171	63	4.4293	115	8.0853	330	23.2012
24	1.6874	64	4.4996	120	8.4368	340	23.9043
25	1.7577	65	4.5699	125	8.7884	350	24.6073
26	1.8280	66	4.6402	130	9.1399	360	25.3104
27	1.8983	67	4.7106	135	9.4914	370	26.0135
28	1.9686	68	4.7809	140	9.8430	380	26.7166
29	2.0389	69	4.8512	145	10.1945	390	27.4196
30	2.1092	70	4.9215	150	10.5460	400	28.1227
31	2.1795	71	4.9918	155	10.8976	410	28.8258
32	2.2498	72	5.0621	160	11.2491	420	29.5288
33	2.3201	73	5.1324	165	11.6006	430	30.2319
34	2.3905	74	5.2027	170	11.9522	440	30.9350
35	2.4607	75	5.2730	175	12.3037	450	31.6380
36	2.5310	76	5.3433	180	12.6553	460	32.3411
37	2.6013	77	5.4136	185	13.0068	470	33.0442
38	2.6717	78	5.4939	190	13.3583	480	33.7487
39	2.7420	79	5.5542	195	13.7099	490	34.4503
40	2.8123	80	5.6246	200	14.0614	500	35.1533

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